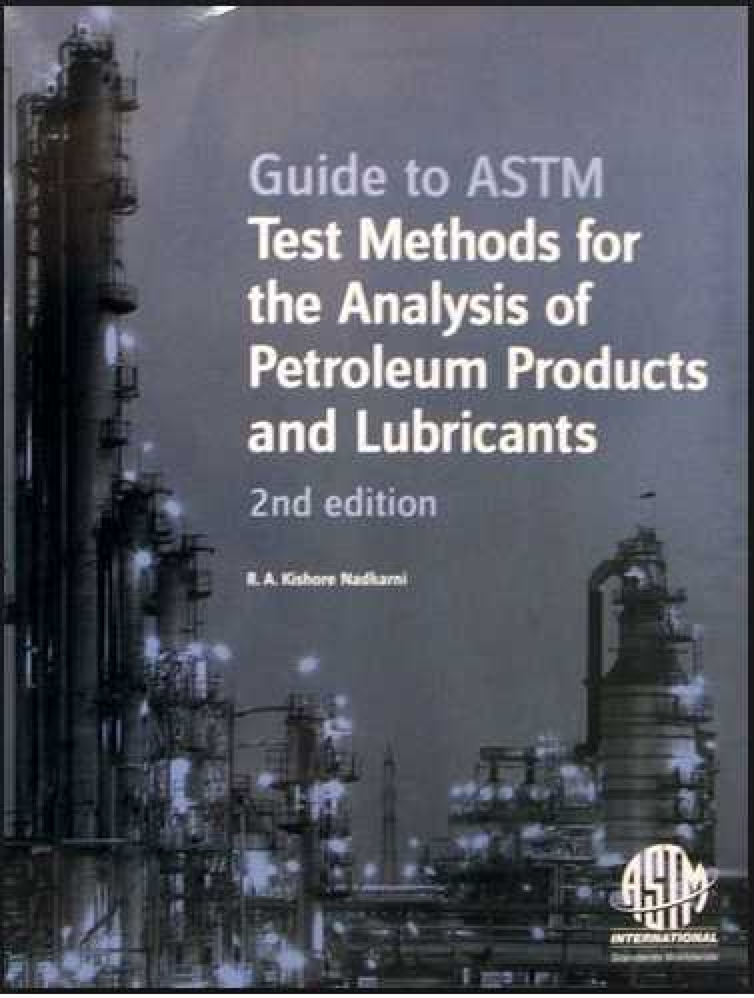

Guide to ASTM Test Methods for the Analysis of Petroleum Products and Lubricants

2nd edition

R. A. Kishore Nadkarni

ASTM INTERNATIONAL

Guide to ASTM Test Methods for the Analysis of Petroleum Products and Lubricants
2nd Edition

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ASTM Stock No. MNL44–2nd

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Printed in U.S.A.

Library of Congress Cataloging-in-Publication Data

Nadkarni, R. A.
Guide to ASTM test methods for the analysis of petroleum products and lubricants / R. A. Kishore Nadkarni–2nd ed.
p. cm. (ASTM manual series; no. mnl44-2nd)
Includes bibliographical references and index.
ISBN 978-0-8031-4274-9
1. Petroleum products–Testing–Standards. 2. Lubrication and lubricants–Testing–Standards. I. Title.
TP691.N33 2007
665.5'380287-dc22
2007022845

Printed in City, State
Month 2007

How To Use This Manual

Table I—lists the test methods with their equivalent IP, ISO, DIN, JIS, and AFNOR designations. The top of each page listing the test summary also refers to these equivalent standards. If you are considering using any standard that has equivalent standards you should refer to them to determine the full scope of each standard and any differences between. Although these standards are listed as equivalent they will not be exactly the same in many cases.

Table II—lists the ASTM test methods alphanumerically by ASTM designation. If you know the ASTM designation, this is the easiest way to find what you need. The top of each page listing the test summary also refers to these equivalent standards.

Foreword

THE PUBLICATION, Guide to ASTM Test Methods for the Analysis of Petroleum Products and Lubricants: 2nd Edition, was sponsored by ASTM Committee D02 on Petroleum Products and Lubricants and edited by R. A. Kishore Nadkarni, Millennium Analytics, Inc., East Brunswick, NJ. This is Manual 44–2nd of ASTM's manual series.

This manual originally published in 2000 has proved to be a useful reference book to technologists and others in the Petroleum Products and Lubricants industry. This enlarged second edition is updated to include ASTM D02 Committee test methods published through the end of 2006. The manual contains descriptions of a total of 585 test methods (an increase of 222 test methods from 363 methods in the first edition) describing a total of about 229 chemical and physical tests used to analyze petroleum products and lubricants (an increase of about 69 properties from about 160 properties described in the first edition).

The author and the publisher hope that this second edition will prove as useful as the first one to the oil industry researchers, analysts, and marketers.

CONTENTS

INTRODUCTION

CRUDE OILS, petroleum products and lubricants are highly complex materials and enormous efforts have been spent by the oil companies throughout the world to characterize their chemical and physical properties with a high degree of precision and accuracy. The explosive growth in the availability of modern analytical instrumentation in the last four to five decades has significantly helped in the task of petroleum products analyses. These modern techniques have largely supplanted the classical "wet chemistry" types of analyses, which were used in the first half of the last century [1, 2]. However, there are still a few areas where some specific analyses need these older techniques.

ASTM Committee DO2 Petroleum Products and Lubricants has since the last century led these efforts to develop more reliable and standard test methods to the point that in all corners of the world ASTM DO2 standards are considered as the final arbitrators of the quality of a petroleum related product. Other national and international standardization bodies such as EI (formerly called IP) in U.K., AFNOR in France, DIN in Germany, JIS in Japan, and ISO have also contributed significantly in developing standard test methods for the analyses of petroleum products. However, many of the latter standards are based on the ASTM DO2 standards.

There are about 580 ASTM test method standards available that involve a variety of analytical techniques to identify or quantify, or both, about 230 chemical and physical properties of crude oils, gasoline, reformulated gasoline, lubricating oils, additives, transmission fluids, lubricating greases, gear oils, aviation fuels, diesel and heating fuels, petroleum waxes, marine fuels, and other specialty petroleum products. These test methods are annually published in the *Annual Book of ASTM Standards* [3]. Additional methods continue to be developed in many oil company laboratories either to improve on the existing methods or to enable determination of other properties. Many of such methods remain proprietary to the oil companies.

An earlier manual on the significance of tests for petroleum products emphasized the rationale of specific tests conducted on different products [4]. However, this valuable manual did not include the details of any tests, but rather discussed the compositions of products and the primary quality characteristics that defined that material's use in applications.

It is certainly not the intention of this author to replace the *Annual Book of ASTM Standards* or the other valuable manual with the current book, but rather to view it as a complementary material for the customer. The purpose of this book is to make available in one handy volume, the essential elements of all analytical tests used to characterize the petroleum products. It is of course critical for the testing laboratory personnel to be fully familiar with all the details of the tests they are performing. But it is also important for non-laboratory personnel to know at least the significance, advantages and limitations of particular tests used to characterize the product quality. Both the suppliers and the customers need to agree on the appropriate product quality specifications, and this can be done only by understanding the pros and cons of these tests. Product specifications not based on realistic testing capabilities can only lead to quality complaints and unhappiness on the part of both suppliers and customers. As such, we expect that this book will prove useful not only to the laboratory personnel, but also to the product specification writers, formulators, process engineers, researchers, and marketing staff in understanding the importance of these tests as well as their limitations, so that sound conclusions can be reached regarding the quality and performance of a company's products.

Table 1 lists other international test methods that are technically equivalent to the ASTM test methods. No claim is being made that all details in ASTM and non-ASTM standards are exactly the same. However, it is expected that if properly followed, both sets should give equivalent results. In an increasingly global marketplace, it is important to be cognizant of such equivalency. Much of this information on the equivalent test methods has been extracted from a compilation that has been prepared by Mr. Tim Berryman and published by the Institute of Petroleum, London. Although at one time leading European national standards organizations such as IP, DIN, and AFNOR produced their own independent standards, lately they are being integrated with the ISO (or more specifically EN-ISO) series standards. The reason for this is that there is a European Union legal requirement to publish all EN standards as the national standards and to withdraw all existing conflicting standards. The vast majority of EN standards *are* actually implementation of the corresponding ISO standards. Table 1 retains the original designations of the European national bodies with which most people are familiar, rather than the new common EN-ISO designations. Again, it is not claimed that the alternate methods are exactly equivalent, but it should be remembered that they may be technically equivalent or technically related. The readers are encouraged to consult the IP publication of Mr. Berryman's compilation for better understanding of the relationship among the test methods. My thanks to Mr. Berryman for permission to include his data here.

All technical information included in this book is based on the Year 2007 editions of the *Annual Book of ASTM Standards* [3]. We plan to update this manual at some frequency depending on how many new or significantly revised test method standards are issued by ASTM Committee DO2 on Petroleum Products and Lubricants. If you notice any errors or omissions, please let us know. We will correct them in future editions.

References

[1] Nadkarni, R. A., Ed., *Modern Instrumental Methods of Analysis of Petroleum Products and Lubricants, ASTM STP 1109*, ASTM, 1991.

[2] Nadkarni, R. A., Ed., *Elemental Analysis of Fuels and Lubricants: Recent Advances and Future Prospects, ASTM STP 1468*, ASTM, 2004.

[3] *Annual Book of ASTM Standards*, Volumes 05.01, 05.02, 05.03, and 05.04 (2006).

[4] Rand, S. J., Ed., *Manual on Significance of Tests for Petroleum Products*, 7th ed., ASTM, West Conshohocken, PA, MNL 1, 2003.

Explanation of Terms

Accuracy—The accuracy of a test is a measure of how close the test result will be to the true value of the property being measured. As such the accuracy can be expressed as the bias between the test result and the true value. However, the absolute accuracy can only be established if the true value is known.

AFNOR—Association Francaise de Normalisation (Paris).

ASTM—American Society for Testing and Materials (U.S.A.).

DIN—Deutsche Institut Fur Normung (Germany).

IP—Institute of Petroleum (U.K.); now called as Energy Institute.

ISO—International Organization for Standardization (Switzerland).

JIS—Japan Industrial Standards (Tokyo).

Precision—The precision of a test method is defined in terms of the variability between test results obtained on the same material, using a specific test method. The precision of a test is usually unrelated to its accuracy. The results may be precise, but not necessarily accurate. Figures 1 to 4[1] depict in a bull's eye analogy the relation between precision and accuracy. Ideal condition would be most precise and most accurate results. Precision is expressed as repeatability and reproducibility.

Repeatability—The "within-laboratory precision" refers to the precision of a test method when the results are obtained by the same operator in the same laboratory using the same apparatus.

Repeatability or repeatability interval of a test (indicated with the letter "*r*") is defined as the maximum permissible difference due to test error between two results obtained on the same material in the same laboratory.

$$r = 2.77 \times \text{standard deviation of test}$$

Most commonly this repeatability interval (r) is statistically defined at the 95 % probability level, meaning that, even in normal conditions, differences between two test results are unlikely to exceed this repeatability interval more than five times in a hundred.

Reproducibility—The "between-laboratory precision" is defined in terms of the variability between test results obtained on the aliquots of the same homogeneous material in different laboratories using the same test method.

The term reproducibility or reproducibility interval (designated as "*R*") is completely analogous to the term repeatability. Only in this case, it is the maximum permissible difference between two results obtained on the same material but now in different laboratories. The statistical definition of reproducibility is along the same lines as above. Therefore differences between two or more laboratories are unlikely to exceed the reproducibility interval more than five times in a hundred.

$$R = 2.77 \times \text{standard deviation of test}$$

Reproducibility is generally higher than repeatability by a factor of 2 to 4.

The repeatability and reproducibility values have very important implications in today's quality conscious market. As the demand for clear product specifications, and hence control over product consistency grows, it is meaningless to establish product specifications that are more restrictive than the reproducibility/repeatability values of the specification test methods.

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[1] Committee on Standards, "Precision and Bias," *ASTM Standardization News*, ASTM, January 1985, p. 45.

TABLE 1—Test Method Equivalence.[a]

Analysis	ASTM	IP	ISO	DIN	JIS	AFNOR
Tag flash point	D 56			51411	K 2580	M07-003
Relative Density of Crude Oils	D 70		3838			
Distillation	D 86	123	3405	51751	K 2254	M07-002
Melting point	D 87	55	3841	51570		T60-114
Cleveland Open Cup flash point	D 92	36	2592	51376	K 2265	T60-118
Pensky-Martens flash point	D 93	34	2719	51758	K 2265	M07-019
Saponification number	D 94	136	6293	51559	K 2503	T60-110
Water distillation	D 95	74	3733	51582	K 2275	T60-113
Pour point	D 97	15	3016	51597	K 2269	T60-105
Drop melting point	D 127	133	6244			T60-121
Sulfur, bomb method	D 129	61		51577		T60-109
Copper corrosion	D 130	154	2160	51759	K 2513	M07-015
Saybolt color	D 156			51411	K 2580	M07-003
Conradson carbon residue	D 189	13	6615	51551	K 2270	T60-116
Cone penetration	D 217	50	2137			T60-132
Specific energy, bomb method	D 240	12		51900		M07-030
Gasoline diluent in used engine oils by distillation	D 322	23				
Reid vapor pressure	D 323	69	3007	51754	K 2258	M41-007
Gum in fuels	D 381	131	6246	51784	K 2261	M07-004
Kinematic viscosity	D 445	71-1	3104	51562	K 2283	T60-100
Kinematic viscometer calibration	D 446	71-2	3105	51562		
Sediment	D 473	53	3735	51789		M07-063
Ash	D 482	4	6245		K 2272	M07-045
Ramsbottom carbon residue	D 524	14	4262			T60-117
Oxidation stability	D 525	40	7536	51780		M07-012
Dropping point	D 566	132	2176			T60-102
Aniline point	D 611	2	2977	51775	K 2256	M07-021
Ignition quality	D 613	41	5165		K 2280	M07-035
Neutral number	D 664	177	6619		K 2501	
Rust	D 665	135	7120	51585	K 2510	T60-151
Wax oil	D 721	158	2908			T60-120
Oxidation stability	D 873	138				M07-013
Sulfated ash	D 874	163	3987	51575	K 2272	T60-143
Foam	D 892	146	6247	51566	K 2518	T60-129
Cone penetration of lubricating greases	D 937	179	2137	51580		T60-119
Congealing point	D 938	76	2207			T60-128
Relative density of crude oils	D 941		3838			
Oxidation stability	D 942	142		51808		
Oxidation characteristics of inhibited mineral oils	D 943		4263			
Interfacial tension-Ring method	D 971		6295			
Neutral number	D 974	139	6618	51558T1	K 2501	T60-112
Water reaction	D 1094	289	6250	51415		M07-050
Bromine number	D 1159	130	3839			
Distillation characteristics at reduced pressures	D 1160		6616			
Refractive index	D 1218		5661	51423T2		
Water washout	D 1264	215	11009	51807T2		
LPG Sampling	D 1265		4257			
Sulfur by lamp	D 1266	107				M07-031
Liquefied petroleum gases (LPG) vapor pressure	D 1267	161	4256	51616		
Corrosive sulfur in electric insulating oils	D 1275		5662			
Density	D 1298	160	3675	51757H	K 2249H	T60-101
Hydrocarbon types	D 1319	156	3837	51791	K 2536	M07-024

TABLE 1—(*Continued.*)

Analysis	ASTM	IP	ISO	DIN	JIS	AFNOR
Needle penetration	D 1321	49		51579		T66-004
Smoke point	D 1322	57	3014	51406	K 2537	M07-028
Water separability	D 1401	412	6614			T60-125
Cone penetration of lubricating greases	D 1403	310	2137			T60-140
Color, ASTM	D 1500	196	2049	51578	K 2580	T60-104
Vanadium in fuel oils	D 1548					M07-027
Sulfur by high temperature	D 1552					M07-025
Density	D 1657	235	3993			M41-008
Karl Fischer water	D 1744		6296			T60-154
Sediment and water	D 1796	75	3734	51793		M07-020
Odor of petroleum wax	D 1833	185				
Copper corrosion in LPG	D 1838	411	6251			M41-010
Residue in LPG	D 2158	317	13757			
Gas chromatography (GC) analysis of LPG	D 2163	264	7941	51619		M41-013
Dropping point lubricating grease	D 2265		6299			
Four ball method	D 2266	239	11008	51350		
Viscosity index calculations	D 2270	226	2909			T60-136
Oxidation stability	D 2274	388	12205			
Particle contamination in aviation fuels by line sampling	D 2276	216				
Freezing point	D 2386	16	3013	51421	K 2276	M07-048
H_2S in LPG	D 2420	401	8819	51855T3		M41-011
Sulfur by Wickbold method	D 2485		4260			
Cloud point	D 2500	219	3015	51597	K 2269	T60-105
Load carrying capacity of lubricating greases	D 2509	326				
Lead, volumetric	D 2547	248	2083			M07-014
Extreme pressure properties	D 2596		11008			
LPG properties	D 2598	432	8973			
Cold cranking simulator	D 2602	350		51377		
Sulfur by X-ray fluorescence	D 2622			51400T6	K 2541	
Electrical conductivity	D 2624	274	6297	51412T2		
Bromine Index	D 2710	299				
Dryness of propane–Valve freeze method	D 2713		13758			
Timken	D 2782	240		51434		
Sulfur by Wickbold combustion	D 2785	243	4260			T60-142
Boiling range by GC	D 2887		3924			
Distillation characteristics with 15-plate column	D 2892		8708			
Base number	D 2896	276	3771		K 2501	
Brookfield viscosity	D 2983	267	9262			T42-011
Sulfur by oxidative microcoulometry	D 3120		16591			
Mercaptan sulfur	D 3227	342	3012		K 2276	M07-022
Lead by atomic absorption spectroscopy (AAS)	D 3237	428				
JFTOT	D 3241	323	6249		K 2276B	M07-051
Acidity of aviation turbine fuel (ATF)	D 3242	354		51558T3		
Sulfur by oxidative microcoulometry	D 3246	373				M07-052
Acid numbers–color indicator titration	D 3339		7537			
Lead by iodine chloride	D 3341		3830			
Metals by atomic absorption (AAS)	D 3605	413	8691	51790T3		
Benzene by GC	D 3606	425				
Hydrogen content of aviation turbine fuels by NMR	D 3701	338				
Sulfonates	D 3712	369				
Seta flash point	D 3828	303	3679			
Water by distillation	D 4006	358	9029			
Water and sediment by centrifuge method	D 4007	359	9030			

TABLE 1—(*Continued.*)

Analysis	ASTM	IP	ISO	DIN	JIS	AFNOR
Alkyl nitrate	D 4046	430	13759			
Phosphorus by colorimetry	D 4047	149	4265			
Density, digital	D 4052	365	12185	51757D	K 2249D	T60-172
Benzene by infrared spectroscopy (IR)	D 4053	429				
Petroleum products—Manual sampling	D 4057		3170			
Petroleum products—Automatic sampling	D 4177		3171			
Sulfur by ND-XRF	D 4294	336	8754			M07-053
Water by potentiometric Karl Fischer titration	D 4377	356	10336			
Aniline point	D 4529	381	3648			
Microcarbon residue	D 4530	398	10370			
Distillation fuel storage stability	D 4625	378				
Metals by AAS	D 4628	308		51391T1		
Nitrogen by chemiluminescence detection	D 4629	379				M07-058
Calculation of cetane index	D 4737		4264			
Base number	D 4739	417	6619		K 2501	
Sediment	D 4870	375	10307			
Metals by wavelength-dispersive X-ray fluorescence (WDXRF)	D 4927	407		51391T2		
Karl Fischer water	D 4928	386	10337			
Doctor test	D 4952	30	5275			
Aluminum and silicon in fuels	D 5184	377	10478	51416		
Vapor pressure	D 5191	394				M07-079
Shear stability index	D 5275	294		51382		
Oxygenates	D 5599	408				
Cloud point by optical detection	D 5771	444				
Cloud point by linear cooling rate	D 5772	445				
Cloud point by constant cooling rate	D 5773	446				
Noack volatility	D 5800	421		51581		
Pour Point of Crude Oils	D 5853	441				
Pour point of crude oils	D 5863	441				
Freezing point	D 5901	434				
Auto-freeze point	D 5972	435				
Particulate matter	D 6217	415	15167			
Test precision calculations	D 6300		4259			
Cold filter plugging	D 6371	309	116			
Friction and wear	D 6425			51834		
Aromatic hydrocarbon	D 6379	436				

TABLE 2—Alphanumeric Index Reference to ASTM Standards by Designation Number.

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D 61	Softening Point of Pitches (Cube-in-Water Method)	230
D 71	Relative Density of Solid Pitch and Asphalt (Displacement Method)	81
D 86	Distillation of Petroleum Products	86
D 87	Melting Point of Petroleum Wax (Cooling Curve)	158
D 88	Saybolt Viscosity Precipitation Number of Lubricating Oils	275
D 91	Precipitation Number of Lube Oils	213
D 92	Flash and Fire Points by Cleveland Open Cup	107
D 93	Flash Point by Pensky-Martens Closed Cup Tester	109
D 94	Saponification Number of Petroleum Products	222
D 95	Water in Petroleum Products and Bituminous Materials by Distillation	286
D 96	Water and Sediment in Crude Oils by Centrifuge Method (Field Procedure)	225
D 97	Pour Point of Petroleum Products	210
D 127	Drop Melting Point of Petroleum Wax Including Petrolatum	159
D 128	Analysis of Lubricating Grease	156
D 129	Sulfur in Petroleum Products (General Bomb Method)	239
D 130	Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test	70
D 156	Saybolt Color of Petroleum Products (Saybolt Chromometer Method)	63
D 187	Burning Quality of Kerosene	48
D 189	Conradson Carbon Residue of Petroleum Products	66
D 217	Cone Penetration of Lubricating Grease	64
D 240	Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter	124
D 287	API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)	120
D 322	Gasoline Diluent in Used Gasoline Engine Oils by Distillation	118
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D 525	Oxidation Stability of Gasoline (Induction Period Method)	195
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D 611	Aniline and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents	24
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D 664	Acid Number of Petroleum Products by Potentiometric Titration	17
D 665	Rust Preventing Characteristics of Inhibited Mineral Oil in the Presence of Water	220
D 721	Oil Content of Petroleum Waxes	188
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D 849	Copper Strip Corrosion by Industrial Aromatic Hydrocarbons	70
D 873	Oxidation Stability of Aviation Fuels (Potential Residue Method)	192
D 874	Sulfated Ash from Lubricating Oils and Additives	34
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D 937	Cone Penetration of Petrolatum	65
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D 1093	Acidity of Distillation Residues of Hydrocarbon Liquids and their Distillation Residues	20
D 1094	Water Reaction of Aviation Fuels	286
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D 1264	Water Washout Characteristics of Lubricating Greases	294
D 1266	Sulfur in Petroleum Products (Lamp Method)	243
D 1267	Gage Vapor Pressure of Liquefied Petroleum Gases	117
D 1298	Density, Relative Density (Specific Gravity) or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method	80
D 1319	Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption	131
D 1321	Needle Penetration of Petroleum Waxes	184
D 1322	Smoke Point of Aviation Turbine Fuels	229
D 1364	Water in Volatile Solvents (Karl Fischer Reagent Titration Method)	292
D 1401	Water Separability of Petroleum Oils and Synthetic Fluids	290
D 1403	Cone Penetration of Lubricating Grease Using One-Quarter and One-Half Scale Cone Equipment	64
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D 1465	Blocking and Picking Points of Petroleum Wax	41
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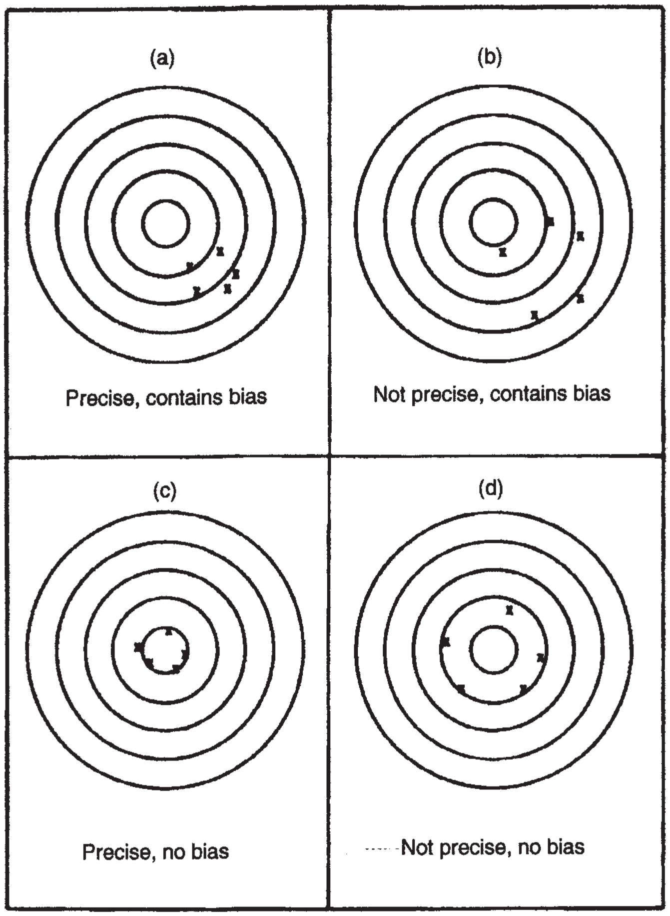

FIG. 1—*Bull's eye analogy.*

ACID NUMBER BY COLOR INDICATOR TITRATION: **D 974** (Equivalent Test Methods: IP 139, ISO 6618, DIN 51558T1, JIS K 2501, and AFNOR T60-112)

EXPLANATION

See Acid number for Test Method D 664 that follows.

Oils such as many cutting oils, rustproofing oils, and similar compounded oils, or excessively dark-colored oils, that cannot be analyzed by this test method due to obscurity of the color-indicator end point, can be analyzed by Test Method D 664. The acid numbers obtained by this color-indicator test method may or may not be numerically the same as those obtained by Test Method D 664.

DEFINITIONS

Acid number—the quantity of base, expressed in milligrams of potassium hydroxide per gram of sample that is required to titrate a sample in this solvent to a green/green-brown end point, using *p*-naphtholbenzein indicator solution.

Strong acid number—the quantity of base, expressed in milligrams of potassium hydroxide per gram of sample, that is required to titrate a hot water extract of the sample to a golden brown end point, using methyl orange solution.

TEST SUMMARY

To determine the acid number, the sample is dissolved in a mixture of toluene and isopropyl alcohol containing a small amount of water, and the resulting single-phase solution is titrated at room temperature with standard alcoholic base or alcoholic acid solution, respectively, to the end point indicated by the color change of the added *p*-naphtholbenzein solution (orange in acid and green-brown in base). To determine the strong acid number, a separate portion of the sample is extracted with hot water and the aqueous extract is titrated with potassium hydroxide solution, using methyl orange as an indicator.

TEST PRECISION

Acid Number	Repeatability	Reproducibility
<0.1	0.03	0.04
0.1 to 0.5	0.05	0.08
0.5 to 1.0	0.08	15%
1.0 to 2.0	0.12	15%

ACID NUMBER BY POTENTIOMETRIC TITRATION: **D 664** (Equivalent Test Methods: IP 177, ISO 6619, and JIS K2501)

EXPLANATION

New and used petroleum products may contain acidic constituents that are present as additives or as degradation products formed during service, such as oxidation products. The relative amount of these materials can be determined by titrating with bases. The acid number is a measure of this amount of acidic substance, in the oil—always under the conditions of the test. The acid number is used as a guide in the quality control of lubricating oil formulations. It is also sometimes used as a measure of lubricant degradation in service. Any condemning limits must be empirically established. Since a variety of oxidation products contribute to the acid number and the organic acids vary widely in corrosion properties, the test cannot be used to predict corrosiveness of an oil under service conditions. No general correlation is known between acid number and the corrosive tendency of oils toward metals.

The test method resolves constituents into groups having weak-acid and strong-acid ionization properties, provided the dissociation constants of the more strongly acidic compounds are at least 1000 times that of the next weaker groups. In new and used oils, the constituents that may be considered to have acidic characteristics include organic and inorganic acids, esters, phenolic compounds, lactones, resins, salts of heavy metals, salts of ammonia and other weak bases, acid salts of polybasic acids, and additional agents such as inhibitors and detergents.

The test method may be used to indicate relative changes that occur in an oil during use under oxidizing conditions regardless of the color or other properties of the resulting oil. There are four test methods for the determination of acid numbers. See Table 3 for comparison of these test methods. Test Method D 4739 is described in the base number section of the manual since it can analyze both acid and base numbers.

DEFINITIONS

Acid number—the quantity of base, expressed in milligrams of potassium hydroxide per gram of sample, required to titrate a sample in the solvent from its initial meter reading to a meter reading corresponding to a freshly prepared nonaqueous basic buffer solution or a well defined inflection point as specified in the test method.

Strong acid number—the quantity of base, expressed as milligrams of potassium hydroxide per gram of sample, required to titrate a sample in the solvent from its initial meter reading to a meter reading corresponding to a freshly prepared nonaqueous acidic buffer solution or a well defined inflection point as specified in the test method.

TEST SUMMARY

The sample is dissolved in a mixture of toluene and isopropyl alcohol containing a small amount of water and titrated potentiometrically with alcoholic potassium hydroxide using

TABLE 3—*Comparison of ASTM Test Methods for Acid Number Determination.*

Area	D 664		D 974	D 3339	D 4739
Significance	Determines acidic constituents (organic/inorganic acids, esters, phenolics, lactones, resins, heavy metal salts, salts of NH_3 and other weak bases, acid salts of poly-basic acids, other inhibitors and detergents).		Same as in D 664.	Same as in D 664. Mainly used for small samples from oxidation test D 943.	Same as in D 664.
	Indicates relative changes in oil under oxidizing conditions; but cannot predict corrosion behavior.		Same as in D 664.	Same as in D 664.	Same as in D 664.
Procedure	Sample dissolved in toluene+IPA+water. Titrated potentiometrically with alcoholic KOH. Inflection or buffer end points.		Same as in D 664 except *p*-naphtholbenzein used as color indicator.	Same as in D 664.	Sample dissolved in toluene+IPA + chloroform + water. Titrated potentiometrically with alcoholic KOH. Inflection or buffer end points.
Scope	Applicable to fresh and used oils. Results may not be same as in D 974 or D 3339.		Applicable to fresh and used oils. Results same as by D 3339, but not D 664.	Applicable to fresh or used oils *for very small samples*. Results same as by D 974, but not D 664.	Applicable to fresh and used oils. Results may not be same as in D 664, D 974 or D 3339.
Limitations	Used oils change on storage. Representative sampling difficult with sediment in used oils.		Difficulties with highly colored oils (use D 664 instead). Used oils change on storage. Representative sampling difficult with sediment in used oils.	Same as in D 974.	Same as in D 644.
Precision[a]	Fresh Oils	Used Oils		r: $0.08\sqrt{X}$	r=10.4%
	v: 0.3 to 0.12 (a)			R: $0.27\sqrt{X}$	R=21.1%
	R: 0.4 to 15% (a)				
	r: 0.044 $(X+1)$	0.117 X			
	R: 0.141 $(X+1)$	0.44 X			

[a] See ASTM test method for details.

a glass indicating electrode and a calomel reference electrode. The meter readings are plotted manually or automatically against the respective volumes of titrating solution and the end points are taken only at well defined inflections in the resulting curve. When no definite inflections are obtained, end points are taken at meter readings corresponding to those found for freshly prepared nonaqueous acidic and basic buffer solutions.

TEST PRECISION

	Repeatability	Reproducibility
Fresh Oils and Additives At Inflection Points	$0.044\ (X+1)$	$0.117\ X$
Used Oils at Buffer End Points	$0.141\ (X+1)$	$0.44\ X$

Where X is the mean result.

ACID NUMBER BY SEMI-MICRO COLOR INDICATOR TITRATION:

D 3339

(Equivalent Tests: IP 431 and ISO 7537)

EXPLANATION

This test method measures the acid number of oils obtained from laboratory oxidation test (Test Method D 943) using smaller amounts of samples than those used in other acid number tests for Test Methods D 664 or D 974. It is applicable for the determination of acids having dissociation constants larger than 10^{-9}. Extremely weak acids or salts whose dissociation and hydrolysis constants, respectively, are 10^{-9} do not interfere. No general relationship between corrosion and this acid number is known. Dark colored oils may be more difficult to analyze by this method because of the difficulty in detecting color change. In such cases Test Method D 664 may be used if sufficient sample is available. The values obtained by Test Methods D 3339 and D 664 may or may not be numerically the same but they should be of the same order of magnitude. However, the values obtained by Test Methods D 3339 and D 974 have been found to be the same within the precision of the two test methods.

TEST SUMMARY

An oil sample is dissolved in a solvent mixture of toluene, isopropyl alcohol, and a small amount of water. The solution is then titrated at room temperature under a nitrogen atmosphere with standard KOH in isopropyl alcohol to the stable green color of the added indicator *p*-naptholbenzein.

TEST PRECISION

Repeatability:	0.05 to 20.0 acid number	$0.08\ (X)^{0.5}$
Reproducibility:	0.05 to 20.0 acid number	$0.27\ (X)^{0.5}$

Where X is the mean acid number of sample.

The test has no known bias.

ACID NUMBER BY SEMI-QUANTITATIVE MICRO DETERMINATION OF ACID NUMBER OF LUBE OILS DURING OXIDATION TESTING:

D 5770

EXPLANATION

This test method provides a means of monitoring the relative oxidation of lubricating oils by measuring changes in acid number, at different time intervals and under various oxidizing test conditions. It gives a semi-quantitative estimate of the acid number using smaller amounts of sample than those required in Test Methods D 664, D 974, or D 3339. This test has specific applications in Test Methods D 943 and D 4871. This test is a micro version of Test Method D 974 and both methods give similar results. The method should, however, not be used as a replacement for higher precision test methods such as D 664 or D 974. It shall not be used to monitor oils in-service. The test is applicable to turbine oils, hydraulic oils, and other circulating oils whose acid numbers fall in the range 0.02 to 1.0 mg of KOH per gram of sample.

TEST SUMMARY

The method is similar to Test Method D 974. A 2.0-mL portion of the solution is titrated with samples using a dropping pipet. The number of drops of sample required to turn the blue-green titration solution to a persistent orange color is noted. A second 2.0-mL portion of the titration solution is then titrated with an acid number reference solution of known acid number the same way as the first time. From the ratio of number of drops required for color change in above two titrations, the acid number is calculated.

TEST PRECISION

In the acid number range of 0.06 to 1.1 mg KOH/g of sample, the repeatability and reproducibility were found to be 0.20× acid number and 0.70× acid number, respectively. The procedure is not known to have any bias.

ACIDITY IN AVIATION TURBINE FUEL:

D 3242

(Equivalent Tests: IP 354 and DIN 51558 T3)

EXPLANATION

Some acids can be present in aviation turbine fuels due either to the acid treatment during the refining process or to naturally occurring organic acids. It's unlikely that significant acid contamination will be present because of many check tests made during the refining process. In any case these trace acid quantities are undesirable because of the possibility of metal corrosion and impairment of water separation characteristics of the fuel.

This test method covers the determination of the acidity in an aviation turbine fuel in the range 0.000 to 0.100 mg KOH/g. It is, however, not suitable for determining significant acid contamination.

TEST SUMMARY

A sample is dissolved in a solvent mixture (toluene plus isopropyl alcohol, and a small amount of water) and under a stream of nitrogen is titrated with standard alcoholic KOH to the color change from orange in acid to green in base via added indicator *p*-naptholbenzein solution.

TEST PRECISION

The test repeatability is 0.0132 $\sqrt{a}$ and test reproducibility is 0.0406 $\sqrt{a}$ where a is the acid number. These data are based on the manual burets only; precision for using automated burets is not known. This test method has no bias.

ACIDITY OF HYDROCARBON LIQUIDS AND THEIR DISTILLATION RESIDUES:

D 1093

EXPLANATION

Some petroleum products are treated with mineral acid during the refining process. Any residual mineral acid in a petroleum product is undesirable. This test makes a qualitative determination of the acidity of hydrocarbon liquids and their distillation residues. The results are qualitative. Basicity determination can also be done by a small change in the procedure (see below).

TEST SUMMARY

A sample is shaken with water and the aqueous layer is tested for acidity using methyl orange indicator (red color). Basicity can be determined using phenolphthalein indicator (pink color) instead of the methyl orange indicator.

TEST PRECISION

This is a pass-fail qualitative test and no precision or bias estimates can be made.

ACTIVE SULFUR IN CUTTING OILS: D 1662

EXPLANATION

This test measures the amount of sulfur available to react with metallic surfaces to form solid lubricating aids at the temperature of the test. The rates of reaction are metal type, temperature, and time dependent. It has not been determined as to how the active sulfur content thus determined may relate to field performance of the cutting fluid.

TEST SUMMARY

A sample is treated with copper powder at 149°C or 300°F. The copper powder is filtered from the mixture. Active sulfur is calculated from the difference between the sulfur contents of the sample, as determined by Test Method D 129, before and after treatment with copper.

TEST PRECISION

Precision is not known at present since a round robin has not been completed.

ACTIVE SULFUR IN FUELS AND SOLVENTS (DOCTOR TEST): D 4952

(Equivalent Tests: IP 30 and ISO 5275)

EXPLANATION

Sulfur present as mercaptans or as hydrogen sulfide in distillate fuels and solvents can attack many metallic and nonmetallic materials in fuel and other distribution systems. A negative result in the Doctor test ensures that the concentration of these compounds is insufficient to cause such problems in normal use. This test is pertinent for petroleum product specifications given in Specification D 235.

TEST SUMMARY

The sample is shaken with sodium plumbite solution, a small quantity of sulfur is added, and the mixture shaken again. The presence of mercaptans or hydrogen sulfide or both is indicated by discoloration of the sulfur floating at the oil-water interface or by discoloration of either of the phases.

TEST PRECISION

Since this is a pass-fail test, no estimates of precision or bias can be made.

ADHESION OF SOLID FILM LUBRICANTS: D 2510

EXPLANATION

Effective solid film lubricant coatings must adhere to surfaces to provide adequate lubrication in applications with restricted access where fluid lubricants cannot easily be replenished. Without this coating adhesion, metal to metal contact results in significant wear of contacting surfaces. Adhesion is critical to the performance of solid film lubricants in applications such as fasteners, bearings, and sliding members in automotive, aircraft, and aerospace hardware.

This test measures the adhesion of solid film lubricant coatings when submitted to contact with water and other fluids.

TEST SUMMARY

The dry solid film lubricant is applied to anodized aluminum panels, immersed in water or other fluids for 24 hours and then wiped dry. A strip of masking tape is pressed onto the panel and removed abruptly. Film removal exposing the surface of the metal panel is the criterion for failure.

TEST PRECISION

This is a qualitative pass-fail test and no estimates of precision or bias can be made.

AIR RELEASE PROPERTIES OF OILS: D 3427

(Equivalent Test Methods: IP 313, ISO 9120, DIN 51381, and AFNOR E48-614)

EXPLANATION

Agitation of lubricating oil with air in equipment such as bearings, couplings, gears, pumps, and oil return lines may produce a dispersion of finely divided air bubbles in the oil. If the residence time in the reservoir is too short to allow the air bubbles to rise to the oil surface, a mixture of air and oil will circulate through the lubricating oil system. This may result in an inability to maintain oil pressure (particularly with centrifugal pumps), incomplete oil films in bearings and gears, and poor hydraulic system performance or failure.

This test measures the time for the entrained air content to fall to the relatively low value of 0.2 % volume under a standardized set of test conditions and hence permits the comparison of the ability of oils to separate entrained air under conditions where a separation time is available. The significance of this test has not been fully established. However, sponginess and lack of sensitivity of the control systems of some turbines may be related to the air release properties of the oil. System design and system pressure are other variables. Currently the applicability of this test method appears to be directed towards turbines manufactured outside the United States. It may not be suitable for ranking oils in applications where residence times are short and gas contents are high.

TEST SUMMARY

Compressed gas is blown through the test oil which has been heated to a temperature of 25, 50, or 75°C. After the gas flow is stopped, the time required for the gas entrained in the oil to reduce in volume to 0.2 % is recorded as the gas bubble separation time. Gas bubble separation time is defined as the number of minutes needed for gas entrained in the oil to reduce in volume to 0.2 % under the conditions of this test and at the specified temperature.

TEST PRECISION

Repeatability:	$0.5\sqrt{\text{mean}}$
Reproducibility:	$1.3\sqrt{\text{mean}}$

ALKYL NITRATE IN DIESEL FUELS: D 4046

(Equivalent Tests: IP 430 and ISO 13759)

EXPLANATION

Alkyl nitrate is added to diesel fuel to improve cetane number. This method can determine 0.03 to 0.30 volume percent of alkyl nitrate in diesel fuel and thus can serve as a basis for judging compliance with specifications covering any alkyl nitrate. The standards used for calibration must contain the same alkyl nitrate ester as the test specimen to be analyzed. Other nitrate esters, inorganic nitrate ions, and nitrogen oxides interfere.

TEST SUMMARY

Simultaneous hydrolysis of ester in 65 % sulfuric acid solution and nitration of *m*-xylenol by the nitric acid liberated is carried out. The resultant nitroxylenol is extracted from the reaction mixture and reacted with NaOH to form the yellow salt. The color is measured spectrophotometrically at 452 nm, and the concentration of alkyl nitrate is determined by reference to the slope of the calibration curve.

TEST PRECISION

Repeatability:	0.017 volume %
Reproducibility:	0.036 volume %

The test method has no known bias.

AMYL NITRATE IN DIESEL FUELS:
D 1839

EXPLANATION

See Test Method D 4046 for details substituting amyl for alkyl nitrate. The interferences are the same as in Test Method D 4046. This method can also be used for determining hexyl nitrate in diesel fuels provided standards containing nitrate esters of primary hexanol are used.

TEST SUMMARY

Hydrolysis of the ester in sulfuric acid and nitration of *m*-xylenol by the nitric acid is simultaneously carried out. The nitroxylenol is extracted from the reaction mixture and is reacted with NaOH to form the yellow sodium salt. Residual diesel fuel is removed by ether extraction. The color is measured spectrophotometrically at 452 nm. The concentration of amyl nitrate is determined by reference to a standard curve.

TEST PRECISION

Both repeatability and reproducibility of this test method is reported to be 0.10 volume percent. This test method has no known bias.

ANALYSIS OF LPG AND PROPANE CONCENTRATES BY GC:
D 2163

EXPLANATION

The component distribution of liquefied petroleum gases (LPG) and propane concentrates is often required as a specification analysis for end-use sale of these materials. Precise compositional data are required to assure uniform quality of the desired reaction products. These data can also be used to calculate physical properties such as relative density, vapor pressure, and motor octane. This test method provides such analyses, and is applicable to analysis of propane, propene, and butane in all concentration ranges from 0.1 % and above.

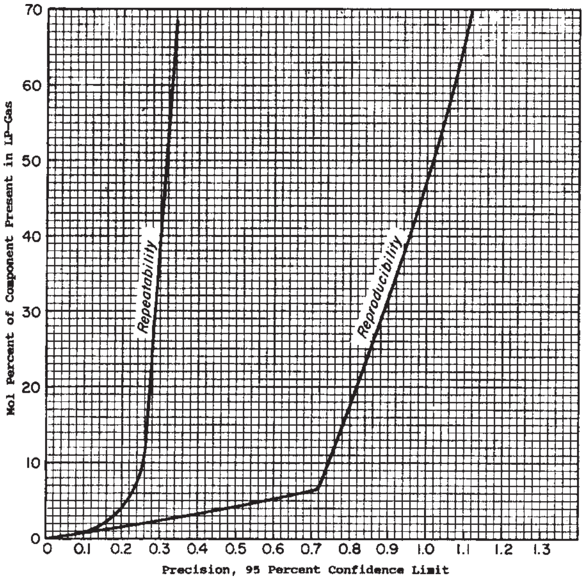

FIG. 2—*Precision of D 2163 Test Method.*

Precision Data for LPG Containing Less Than 50 % Propene.

Concentration Range of Components, mol %	Repeatability	Reproducibility
0 to 70	Use repeatability curve in Fig. 2(*a*)	Use reproducibility curve in Fig. 2(*a*)
Above 70	0.2	1 % of amount present

Precision Data for Propene Concentrates.

Compound	Concentration, mol %	Repeatability	Reproducibility
Ethane	0.0 to 0.1	0.02	0.04
	0.2	0.05	0.06
Propene	70 to 77	0.38	1.5
	93 to 95	0.34	1.0
Propane	5 to 7	0.33	1.0
	22 to 29	1.0	1.7
Butanes	0.0 to 0.1	0.04	0.08
	0.5	0.04	0.2
	0.6	0.1	0.3
	1	0.1	0.5
Butenes	0.2	0.07	0.2

TEST SUMMARY

Components in a sample of LPG are physically separated using gas chromatograph and compared to corresponding components separated under identical operating conditions from a reference standard mixture of known composition or from use of pure hydrocarbons. The chromatogram of the sample is interpreted by comparing peak heights or areas with those obtained on the reference standard mixture of pure hydrocarbons.

TEST PRECISION

No statement of bias can be made since no accepted reference material suitable for this analysis is available.

ANILINE AND MIXED ANILINE POINT: D 611

(Equivalent Tests: IP 2, ISO 2977, DIN 51775, and AFNOR M07-02)

EXPLANATION

The aniline (or mixed aniline) point helps in characterization of pure hydrocarbons and in their mixtures. Aromatics exhibit the lowest and paraffins the highest values. Cycloparaffins and olefins exhibit values between these two extremes. In homologous series the aniline point increases with increasing molecular weight. Aniline point measurement is most often used to estimate the aromatic content of mixtures.

TEST SUMMARY

There are five methods of analysis in this test method:

Method A is used for transparent samples with an initial boiling point above room temperature and where the aniline point is below the bubble point and above the solidification point of the aniline-sample mixture,

Method B, a thin film method, is suitable for samples too dark for testing by Method A,

Methods C and D are for samples that may vaporize appreciably at the aniline point,

Method D is particularly suitable where only small quantities of sample are available, and

Method E uses an automatic apparatus suitable for the range covered by Methods A and B.

TEST PRECISION

	Repeatability	Reproducibility
Aniline Point of:		
Clear lightly colored samples	0.16 (0.3)	0.5 (0.9)
Moderately to very dark samples	0.3 (0.6)	1.0 (1.8)
Mixed Aniline Point of:		
Clear lightly colored samples	0.16 (0.3)	0.7 (1.3)
Moderately to very dark samples	0.3 (0.6)	1.0 (1.8)

All values are in °C (°F).

Bias of this test method has not been developed.

APPARENT VISCOSITY USING HTHS CAPILLARY VISCOMETERS:

Several different configurations of capillary viscometers have been successfully used for measuring the viscosity of engine oils at the high shear rates and high temperatures that occur in engines. There are at least five methods available for this determination.

EXPLANATION

Viscosity is an important property of a fluid lubricant. Many common petroleum lubricants are non-Newtonian; their viscosity varies with shear rate. The viscosity of all fluids varies with temperature. The viscosity of lubricants is to be measured at or near the conditions of shear rate and temperature that they will experience in service. The conditions of shear rate and temperature of these test methods are thought to be representative of those in the bearings of automotive engines in severe service.

APPARENT VISCOSITY BY CAPILLARY VISCOMETER AT HIGH TEMPERATURE HIGH SHEAR:

D 4624

TEST SUMMARY

This test method covers the laboratory determination of the viscosity of engine oils at 150°C and $1 \times 10^6\ \mathrm{s}^{-1}$ using capillary viscometers. Viscosities are determined directly from calibrations that have been established with Newtonian oils in the range from 2 to 7 mPa·s.

Viscosity is determined from measurements of the relationship between pressure drop and flow rate through a capillary under the desired conditions. Two types of capillary viscometers are recognized by this test method:

Constant Pressure Viscometer—in which the pressure is set to a desired shear stress and the resulting flow rate is measured.

Constant Flow-Rate Viscometer—in which the flow rate is set to give a desired shear rate and the resulting pressure drop through the capillary is measured.

For viscometers of both types, the capillary is first calibrated by establishing the relationship between viscosity and flow rate or between viscosity and pressure drop for a series of Newtonian oils by the same procedure and under the same conditions used for test oils.

This test method determines the viscosity at 150°C and a shear rate of $10^6\ \mathrm{s}^{-1}$. For constant pressure viscometers, the viscosity at $10^6\ \mathrm{s}^{-1}$ is determined by interpolation from viscosities measured at several shear stresses. Constant flow-rate viscometers usually can be set for a flow rate that will be at or very close to a shear rate of $10^6\ \mathrm{s}^{-1}$. Operating procedures are highly dependent on the design of each particular viscometer.

D 4683

TEST SUMMARY

This test method covers the laboratory determination of the viscosity of engine oils at 150°C and $1 \times 10^6\ \mathrm{s}^{-1}$ shear rate using a tapered bearing simulator-viscometer (TBS Viscometer) equipped with a refined thermoregulator system. Older TBS units not so equipped must use Test Method D 4683-87.

The Newtonian calibration oils used to establish this test method cover the range from approximately 1.5 to 5.6 cP (mPa·s) at 150°C. The non-Newtonian reference oil used to establish this test method has a viscosity of approximately 3.5 cP (mPa·s) at 150°C and a shear rate of $1 \times 10^6\ \mathrm{s}^{-1}$.

Applicability to petroleum products other than engine oils has not been determined in preparing this test method.

A motor drives a tapered rotor that is closely fitted inside a matched stator. The rotor exhibits a reactive torque response when it encounters a viscous resistance from an oil that fills the gap between the rotor and stator. Two oils, a calibration oil and non-Newtonian reference oil, are used to determine the gap distance between the rotor and stator so that a shear rate of 1×10^6 s^{-1} is maintained. Additional calibration oils are used to establish the viscosity/torque relationship that is required for the determination of the apparent viscosity of test oils at 150°C.

D 4741

TEST SUMMARY

This test method covers the laboratory determination of the viscosity of oils at 150°C and 1×10^6 s^{-1} using Ravenfield high shear rate tapered-plug viscometer models BE or BS. This test method may readily be adapted to other conditions if required.

Newtonian calibration oils are used to adjust the working gap and for calibration of the apparatus. These calibration oils cover a range from approximately 1.8 to 5.9 cP (mPa·s) at 150°C. This test method should not be used for extrapolation to higher viscosities than those of the Newtonian calibration oils used for calibration of the apparatus.

APPARENT VISCOSITY USING COLD CRANKING SIMULATOR:
D 5293

EXPLANATION

The cold cranking simulator (CCS) apparent viscosity of automotive engine oils correlates with low temperature engine cranking. CCS apparent viscosity is not suitable for predicting low temperature flow to the engine oil pump and oil distribution system. Because the CRC L-49 test is much less precise and standardized than the CCS procedures, CCS apparent viscosity need not accurately predict the engine cranking behavior of an oil in a specific engine. However, the correlation of CCS apparent viscosity with average L-49 engine cranking results is satisfactory.

This test method covers the laboratory determination of apparent viscosity of engine oils at temperatures between −5 and −30°C at shear stresses of approximately 50 000 to 100 000 Pa and shear rates of approximately 10^5 to 10^4 s^{-1} and viscosities of approximately 500 to 10 000 mPa·s. The results are related to engine-cranking characteristics of engine oils.

TEST SUMMARY

An electric motor drives a rotor that is closely fitted inside a stator. The space between the rotor and stator is filled with oil. Test temperature is measured near the stator inner wall and maintained by regulated flow of refrigerated coolant through the stator. The speed of the rotor is calibrated as a function of viscosity. Test oil viscosity is determined from this calibration and the measured rotor speed.

TEST PRECISION

Repeatability:	5.4 % of Mean
Reproducibility:	8.9 % of Mean

APPARENT VISCOSITY AT HIGH TEMPERATURE HIGH SHEAR BY MULTICELL CAPILLARY VISCOMETER:
D 5481

TEST SUMMARY

This test method covers the laboratory determination of high-temperature high-shear (HTHS) viscosity of engine oils at a temperature of 150°C using a multicell capillary viscometer containing pressure, temperature, and timing instrumentation. The shear rate for this test method corresponds to an apparent shear rate at the wall of 1.5 million reciprocal seconds (1.5×10^6 s^{-1}). This shear rate has been found to decrease the discrepancy between this test method and other high-temperature high-shear test methods used for engine oil specifications. Viscosities are determined directly from calibrations that have been established with Newtonian oils with viscosities from 2 to 7 mPa·s at 150°C.

The viscosity of the test oil in any of the viscometric cells is obtained by determining the pressure required to achieve a flow rate corresponding to an apparent shear rate at the wall of $1.5 \times 10^6\ s^{-1}$. The calibration of each cell is used to determine the viscosity corresponding to the measured pressure.

Each viscometric cell is calibrated by establishing the relationship between pressure and flow rate for a series of Newtonian oils of known viscosity.

HTHS TESTS PRECISION

Test Method	Repeatability	Reproducibility
D 4624	1.05	1.08
D 4683	2.3 % of Mean	3.6 % of Mean
D 4741	2.8 % of Mean	5.0 % of Mean
D 5481	1.6 % of Mean	5.4 % of Mean

APPARENT VISCOSITY OF LUBRICATING GREASES:
D 1092

EXPLANATION

Apparent viscosity versus shear rate information is useful in predicting pressure drops in grease distribution systems under steady-state flow conditions at constant temperature. This test covers measurements in the temperature range from −53 to 37.8°C(−65 to 100°F). The measurements are limited to the range from 25 to 100 000 poises at $0.1\ s^{-1}$ and 1 to 100 at poises at $15\,000\ s^{-1}$. At very low temperatures the shear rate may be reduced because of the great force required to force grease through the smaller capillaries. Precision has not been determined below $10\ s^{-1}$.

TEST SUMMARY

The sample is forced through a capillary by means of a floating piston actuated by the hydraulic system. From the predetermined flow rate and the force developed in the system, the apparent viscosity is calculated by means of Poiseuille's equation. A series of eight capillaries and two pump speeds are used to determine the apparent viscosity at 16 shear rates. The results are expressed as a log-log plot of apparent viscosity versus shear rate.

TEST PRECISION

Sample	Temperature, °F	Repeatability	Reproducibility
Smooth, NLGI 2, Diester	−65	7	12
Smooth, NLGI 2, SAE 20 Oil	77	6	19
Fibrous, NLGI 1, SAE 20 Oil	77	6	23
Viscous, NLGI 1, SAE 90 Oil	77	7	30

The repeatability and reproducibility values are as percent of mean.

The bias of this test method has not been determined.

APPARENT VISCOSITY YIELD STRESS AND APPARENT VISCOSITY AT LOW TEMPERATURE:
D 4684

EXPLANATION

When an engine oil is cooled, the rate and duration of cooling can affect its yield stress and viscosity. In this laboratory test an engine oil is slowly cooled through a temperature range where wax crystallization is known to occur, followed by relatively rapid cooling to the final test temperature. These laboratory test results have predicted as failures the known engine oils that have failed in the field due to the lack of oil pumpability. These documented field failing oils have all consisted of oils normally tested at −25°C. These field failures are believed to be the result of the oil forming a gel structure that results in excessive yield stress or viscosity of the engine oil, or both.

This test method covers the measurement of the yield stress and viscosity of engine oils after cooling at controlled rates over a period exceeding 45 h to a final test temperature between −15 and −30°C. The applicability to petroleum products other than engine oils has not been determined.

TEST SUMMARY

An engine oil sample is held at 80°C and then cooled at a programmed cooling rate to a final test temperature. A low torque is applied to the rotor shaft to measure the yield stress. A higher torque is then applied to determine the apparent viscosity of the sample.

TEST PRECISION

Since yield stress is a pass-fail test, no precision data are available.

Apparent Viscosity Test

Reproducibility,	Repeatability,	
Temperature, °C	Percent of Mean	Percent of Mean
−15	4.2	8.4
−20	7.3	12.1
−25	11.7	17.5
−30	9.3	18.4
−35	20.7	21.5

APPLIED COATING WAX IN CORRUGATED BOARD FACING:

D 3522

EXPLANATION

Wax treatment is used to improve resistance to moisture damage of corrugated fiberboard shipping containers. This commonly involves a light wax saturation applied to the medium and facings, followed by a curtain coating or roll coating operation applying wax to the surface. This test method measures weight of wax that has been applied and the amount of impregnating wax in the same facing. The method assumes that the major portion of the molten coating applied will congeal and remain on the surface without undue migration into the fibrous structure of the medium. This test method is especially applicable to board that has a coated surface and also contains wax saturation within the facing structure.

The amount of surface wax on board that may or may not contain impregnating wax within its structure may be determined alternatively by Test Method D 3521. If it is known that the specimen has coating wax only, with no internal impregnating wax, the total coating wax applied may be determined by Test Method D 3344.

TEST SUMMARY

The coated facing is peeled from the medium and then split into two layers, one bearing the coating on waxed fibers and one containing the waxed fibers only. The layers are extracted separately, collecting both fibers and wax, leading to a calculation of the applied surface coating wax and the amount of impregnating wax.

TEST PRECISION

	Repeatability	Reproducibility
For impregnating wax content in facing	18 %	20 %
For applied coating wax on facing	5 %	6 %

This method has no bias because the value can be defined only in terms of a test method.

AROMATICS AND POLYNUCLEAR AROMATICS IN DIESEL AND AVIATION TURBINE FUELS BY SFC:

D 5186

EXPLANATION

The aromatic hydrocarbon content of motor diesel fuels affects their cetane number and exhaust emissions. The aromatic hydrocarbon and the napthalene content of aviation turbine fuels affects their combustion characteristics and smoke forming tendencies. These properties are included in the aviation turbine fuel Specification D 1655. U.S. EPA regulates aromatic content, and California Air Resources Board (CARB) regulates the total aromatics and polynuclear aromatics hydrocarbon contents of motor diesel fuels.

This test method is applicable to materials in the boiling range of the motor diesel fuels and is unaffected by fuel coloration. Aromatics concentration in the range 1 to 75 mass %, and polynuclear aromatic hydrocarbons in the range 0.5 to 50 mass % can be determined by this test method.

The results obtained by this method are statistically more precise than those obtained by Test Method D 1319 for diesel fuels and aviation turbine fuels. This test method is also quicker. Results from this test method for total polynuclear aromatic hydrocarbons are also expected to be at least as precise as those of Test Method D 2425.

TEST SUMMARY

A small aliquot of the fuel sample is injected onto a packed silica adsorption column and eluted using supercritical carbon dioxide mobile phase. Mono- and poly-nuclear aromatics in the sample are separated from nonaromatics and detected using a flame ionization detector. The detector response to hydrocarbons is recorded throughout the analysis time. The chromatographic areas corresponding to the mono-, polynuclear, and nonaromatic components are determined and the mass percent content of each of these groups is calculated by area normalization.

TEST PRECISION

Component	Repeatability	Reproducibility
Total Aromatics	$0.61(X)^{0.23}$	$0.75\ (X)^{0.23}$
Polynuclear Aromatics where X is <5 mass %	$0.61(X)^{0.16}$	$0.47\ (X)^{0.45}$
Polynuclear Aromatics where X is >10 mass %	$0.61\ (X)^{0.13}$	$1.77\ (X)^{0.50}$

AROMATICS IN FINISHED GASOLINE BY GAS CHROMATOGRAPHY:
D 4420

EXPLANATION

A knowledge of the amount of aromatics in gasoline is helpful in assessing the product quality and in evaluating the health hazard to persons handling and using gasoline. This test method determines benzene, toluene, C_8, C_9, and heavier aromatics, and total aromatics in finished motor gasoline and gasoline blending components. It has not been determined whether this method is applicable to gasolines containing oxygenates such as ethers and alcohols. Following concentration ranges, in liquid volume percent, can be determined by this test method:

Benzene 0.1 to 5; toluene 1 to 20; C_8 3 to 25; C_9 and heavier 5 to 30; and total aromatics 10 to 80.

TEST SUMMARY

A two column chromatographic system connected to a dual filament thermal conductivity detector (or two single filament detectors) is used. Sample is injected into the column containing a polar liquid phase. The nonaromatics are directed to the reference side of the detector and vented to the atmosphere as they elute. The column is backflushed immediately before the elution of benzene, and the aromatic portion is directed into the second column containing a nonpolar liquid phase. The aromatic components elute in the order of their boiling points and are detected on the analytical side of the detector. Immediately after the C_8 aromatics have eluted, the flow through the nonpolar column is reversed to backflush the C_9 and heavier aromatics from the column through the detector. Quantitation is done from the measured areas of the recorded aromatic peaks by utilizing factors obtained from the analysis of a blend of known aromatic content.

TEST PRECISION

Component	Repeatability	Reproducibility
Benzene	$0.171\ X$	$0.408\ X$
Toluene	$0.089\ X$	$0.156\ X$
C_8 Aromatics	$0.121\ X$	$0.190\ X$
C_9+Aromatics	$0.092\ X$	$0.196\ X$
Total Aromatics	$0.078\ X$	$0.141\ v$

Where X is the measured value.

AROMATICS IN FINISHED GASOLINE BY GC: D 5580

EXPLANATION

To reduce the ozone reactivity in toxicity of automotive evaporative and exhaust emissions regulations limiting the concentration of benzene and the total aromatic content of finished gasoline have been established. This test method can be used for this purpose. The method is also applicable to gasolines containing oxygenates such as alcohols and ethers as additives, since it has been found that they do not interfere with the analysis of benzene and other aromatics. Aromatics in the following concentration range can be determined: benzene 0.1 to 5; toluene 1 to 15; individual C_8 aromatics 0.5 to 10; total C_9 and heavier aromatics 5 to 30; and total aromatics 10 to 80 liquid volume %. The aromatic hydrocarbons are separated without interference from other hydrocarbons in the finished gasoline. For C_8 and C_9 and heavier aromatics some interference is expected from nonaromatic hydrocarbons.

TEST SUMMARY

A two column chromatographic system equipped with a column switching valve and a flame ionization detector is used. The sample is doped with an appropriate internal standard such as 2-hexanone. The flame ionization detector response, proportional to the concentration of each component, is used to calculate the amount of aromatics that are present with reference to the internal standard.

TEST PRECISION

Component	Range	Repeatability	Reproducibility
Benzene	0.14–1.79	$0.0265\ (X^{0.65})$	$0.1229\ (X^{0.65})$
Toluene	2.11–10.08	$0.0301\ (X^{0.5})$	$0.0926\ (X^{0.5})$
Ethylbenzene	0.57–2.65	0.029	0.163
p/*m* Xylene	2.06–9.59	0.071	0.452
o-Xylene	0.77–3.92	$0.0296\ (X^{0.5})$	$0.1168\ (X^{0.5})$
C_9+Aromatics	8.32–25.05	$0.0145\ (X+5.157)$	$0.070\ (X+5.157)$
Total Aromatics	16.34–49.07	$0.0899\ (X^{0.5})$	$0.2851\ (X^{0.5})$

Where range is in mass %, and X is mass %.

AROMATICS IN FINISHED GASOLINE BY GC-FTIR: D 5986

EXPLANATION

To assess product quality and meet new fuel regulations, it is necessary to have methods to determine oxygenates, benzene, and aromatic content of gasoline. This method can be used for gasolines that contain oxygenates such as alcohols and ethers as additives. They do not interfere with the benzene and other aromatics by this method. It is applicable in the following concentration range in volume %: 0.1 to 20 per component for ethers and alcohols; 0.1 to 2 for benzene; 1 to 15 for toluene; 10 to 40 for total C_6 to C_{12} aromatics. The method has not been tested for refinery individual process streams such as reformates, fluid catalytic cracking napthas, etc., used in blending of gasolines.

TEST SUMMARY

The sample is injected through a cool on-column injector into a gas chromatograph equipped with a methylsilicone WCOT column interfaced to a FT-IR instrument. Mixtures of specified pure oxygenates and aromatic hydrocarbons are used for calibration. Multipoint calibrations consisting of at least five levels and bracketing the concentration of the specified individual aromatics is required. Unidentified aromatic hydrocarbons present that have not been specifically calibrated for, are quantitated using the response factor of 1,2,3,5-tetramethylbenzene.

TEST PRECISION[1]

Range and Repeatability

Component	Range (Mass/Volume %)	Repeatability	Reproducibility
Aromatics, volume %	13–41	0.55	1.65
Aromatics, mass %	16–49	$0.23X^{0.3333}$	$0.69X^{0.3333}$
Benzene, volume %	0.1–2	$0.0099\ (X+0.6824)$	$0.054\ (X+0.68)$
Benzene, mass %	0.1–2	$0.012\ (X+0.48)$	$0.063\ (X+0.48)$
Toluene, volume %	2–9	0.10	0.23
Toluene, mass %	2–10	0.057	0.20
1-Butanol, mass %	0.5–1	0.082	0.12
1-Propanol, mass %	0.2–1	0.050	0.078
2-Butanol, mass %	0.6–3	0.081	0.45
2-Propanol, mass %	1–2	0.073	0.10
DIPE, mass %	0.3–2	$0.026X^{0.4}$	$0.066X^{0.4}$
ETBE, mass %	1–18	$0.66X^{0.5}$	$0.19X^{0.5}$
Ethanol, mass %	1–12	$0.052X^{0.5}$	$0.11X^{0.5}$
Isobutanol, mass %	0.1–2.0	0.057	0.12
Methanol, mass %	1–5	0.035	0.45
MTBE, mass %	1–15	$0.032X^{0.5}$	$0.017X^{0.6}$
TAME, mass %	1–18	$0.068X^{0.6}$	$0.015X^{0.6}$
tert-Butanol, mass %	1–2	0.051	0.18

[1] This table is a combination of Tables 10 and 11 from Test Method D 5986.

AROMATICS IN GASOLINES BY GAS CHROMATOGRAPHY-MASS SPECTROMETRY (GC-MS): **D 5769**

EXPLANATION

Test methods to determine benzene and the aromatics in gasoline are necessary to assess the product quality and to meet fuel regulations. This test method can be used for gasolines that contain oxygenates such as alcohols and ethers as additives. They do not interfere with the analysis of benzene and other aromatics by this test method. The method is applicable in the concentration range of liquid volume percent:

Benzene 0.1 to 4; toluene 1 to 13; and total C_6 to C_{12} aromatics 10 to 42.

This test method has not been tested on individual hydrocarbon process streams in a refinery, such as reformates, fluid catalytic cracked napthas, etc., used in the blending of gasoline.

TEST SUMMARY

The sample is injected either through the capillary splitter port or a cool-on-column injector into a gas chromatograph equipped with a dimethylpolysiloxane WCOT column interfaced to a fast scanning mass spectrometer. The capillary column is interfaced directly with the mass spectrometer or by way of an open split interface or other appropriate device. Calibration is done with mixtures of specified pure aromatic hydrocarbons. A multipoint calibration consisting of at least five levels and bracketing the expected concentrations of the specified individual aromatics is required. Specified deuterated hydrocarbons are used as the internal standards.

TEST PRECISION

Component	Range (Volume Percent)	Repeatability	Reproducibility
Benzene	0.09–4.0	0.046 ($X^{0.67}$)	0.221 ($X^{0.67}$)
Toluene	1.0–1.3	0.117 ($X^{0.40}$)	0.695 ($X^{0.40}$)
Total Aromatics	9–42	0.0761 ($X^{0.75}$)	0.244 ($X^{0.75}$)

Where X is the volume % of the component.

Bias of this test method is not known.

AROMATICS IN HYDROCARBON OILS BY HIGH RESOLUTION NUCLEAR MAGNETIC RESONANCE (HR-NMR):

D 5292

EXPLANATION

Aromatic content is a key property of hydrocarbon oils and can affect a variety of properties including boiling range, viscosity, stability, and compatibility of oils with polymers. Existing methods for this work use physical measurements and need suitable standards. This method does not need such standards and is applicable to a wide range of hydrocarbon oils that are completely soluble in chloroform and carbon tetrachloride at ambient temperature. The data obtained by this method can be used to evaluate changes in aromatic contents of hydrocarbon oils due to process changes. Applicable samples include kerosenes, gas oils, mineral oils, lubricating oils, coal liquids, and other distillates. The detection limit is about 0.1 mol % for aromatic hydrogen and 0.5 mol % for aromatic carbon atoms using pulse Fourier transform spectrometers. Continuous wave spectrometers measure only aromatic hydrogen content with a detection limit of 0.5 mol %. This test method is not applicable to samples containing more than 1 mass % olefinic or phenolic compounds.

TEST SUMMARY

Hydrogen NMR spectra are obtained on sample solutions in either chloroform or carbon tetrachloride using a continuous wave or pulse FT high resolution NMR spectrometer. Carbon NMR spectra are obtained on the sample solution in chloroform-*d* using a pulse FT high resolution NMR. Tetramethylsilane is preferred as an internal reference.

TEST PRECISION

Analysis	Repeatability	Reproducibility
Aromatic *H* content	0.32 $X^{0.5}$	0.49 $X^{0.5}$
Aromatic *C* content	0.59 $X^{0.5}$	1.37 $X^{0.5}$

For pure hydrocarbon compounds analyzed by this test method no bias was found.

ASH IN COAL TAR AND PITCH:

D 2415

EXPLANATION

This test method determines the amount of inorganic matter in the tar and pitch samples as ash.

TEST SUMMARY

The sample is carefully volatilized and burned in a muffle furnace or by other suitable means, after which the carbonaceous residue is completely oxidized and the remaining ash is stabilized at 900°C in the muffle furnace.

TEST PRECISION

Repeatability:	0.01
Reproducibility:	0.03

This test method has no bias.

ASH IN PETROLEUM COKE:
D 4422

EXPLANATION

The ash content is one of the properties used to evaluate petroleum coke and indicates the amount of undesirable residue present. Acceptable ash content varies with the intended use.

Preparation and testing of the analysis sample must neither remove nor add ash. Improper dividing, sieving, and crushing equipment, and some muffle furnace lining material can contaminate the sample. High sulfur content of the furnace gases, regardless of the source of the sulfur, can react with an alkaline ash to produce erratic results. The furnace must be swept with air to achieve oxidation and to decrease the sulfur content of the gases.

TEST SUMMARY

A sample of petroleum coke is dried, ground, and ashed in a muffle furnace at 700 to 775 °C.

TEST PRECISION

Repeatability:	0.02
Reproducibility:	0.06

Bias depends on the conformance to the empirical conditions of the test. The ash content must not be understood to be the same as the mineral content of the petroleum coke.

ASH FROM PETROLEUM PRODUCTS:
D 482
(Equivalent Test Methods: IP 4, ISO 6245, JIS K 2272, and AFNOR M07-045)

EXPLANATION

Ash in a petroleum product can result from oil or water-soluble metallic compounds or from extraneous solids such as dirt and rust. This test method is not valid for petroleum products containing ash forming additives, including certain phosphorus compounds, or lubricating oils containing lead, or used engine crankcase oils. In certain types of samples, for example, distillate oils, all of the ash forming metals may not be quantitatively retained in the ash. In all such cases sulfated ash procedure in Test Method D 874 should be used.

TEST SUMMARY

The sample in a suitable vessel is ignited and burned until only ash and carbon remain. This carbonaceous residue is further converted to ash by heating in a muffle furnace at 775°C, cooled and weighed.

TEST PRECISION

Ash, Weight Percent	Repeatability	Reproducibility
0.0001 to 0.079	0.003	0.005
0.080 to 0.180	0.007	0.024

ASH SULFATED ASH FROM LUBRICATING OILS AND ADDITIVES:

D 874

(Equivalent Test Methods: IP 163, ISO 3987, DIN 51575, JIS K 2272, and AFNOR T60-143)

EXPLANATION

Sulfated ash is the residue remaining after the sample has been carbonized, and the residue subsequently treated with sulfuric acid and heated to constant weight.

The sulfated ash may be used to indicate the concentration of known metal-containing additives in new oils. When phosphorus is absent, barium, calcium, magnesium, sodium and potassium are converted to their sulfates and tin (stannic) and zinc to their oxides. Sulfur and chlorine do not interfere, but when phosphorus is present with metals, it remains partially or wholly in the sulfated ash as metal phosphates. Since zinc sulfate slowly decomposes to its oxide at the ignition temperature specified in the method, samples containing zinc may give variable results unless the zinc sulfate is completely converted to the oxide. Magnesium does not react the same as other alkali metals in this test. If magnesium additives are present, the data should be interpreted with caution. Samples containing molybdenum may give low results because molybdenum compounds may not be fully recovered at the temperature of ashing.

Application of this test method to sulfated ash levels below 0.02 % is restricted to oils containing ashless additives. The lower limit of the method is 0.005 % sulfated ash. This test method is not intended for the analysis of used engine oils or oils containing lead. Neither is it recommended for the analysis of nonadditive lubricating oils, for which Test Method D 482 should be used. Because of various inter-element interferences discussed above, experimentally obtained sulfated ash values may differ from sulfated ash values calculated from elemental analysis. The formation of such nonsulfated species is dependent on the temperature of ashing, time ashed, and the composition of the metal compounds present in the oils. Hence, sulfated ash requirements generally should not be used in product specifications without a clear understanding between the buyer and the seller regarding the unreliability of a sulfated ash value as an indicator of the total metallic compounds.

TEST SUMMARY

The sample is ignited and burned until only ash and carbon remain. After cooling, the residue is treated with sulfuric acid and heated at 775°C until oxidation of carbon is complete. The ash is then cooled, re-treated with sulfuric acid, and heated at 775°C to constant weight.

Sulfated Ash, Weight Percent	Repeatability	Reproducibility
0.005 to 0.10	$0.047X^{0.85}$	$0.189X^{0.85}$
0.11 to 25	$0.060X^{0.75}$	$0.142X^{0.75}$

Where X is average of two results.

ASPHALTENES (HEPTANE INSOLUBLES) IN CRUDE PETROLEUM AND PRODUCTS:

D 6560

EXPLANATION

Asphaltenes in crude oils may give problems during storage and handling if the suspension of asphaltene molecules is disturbed through excess stress or incompatibility. They are also the last molecules in a product to combust completely, and thus may be one indicator of black smoke propensity. Their composition normally includes a disproportionately high quantity of the sulfur, nitrogen, and metals present in the crude petroleum or petroleum product.

This test method gives a procedure for the determination of the heptane insoluble asphaltene content of gas oil, diesel fuel, residual fuel oils, lubricating oil, bitumen, and crude petroleum that has been topped to an oil temperature of 260°C. Oils containing additives may give erroneous results.

TEST SUMMARY

A portion of the sample is mixed with heptane and the mixture heated under reflux, and the precipitated asphaltenes, waxy substances, and inorganic material are collected on a filter paper. The waxy substances are removed by washing with hot heptane in an extractor.

After removal of the waxy substances, the asphaltenes are separated from the inorganic material by dissolution in hot toluene, the extraction solvent is evaporated, and the asphaltenes weighed.

TEST PRECISION

Repeatability	0.1 A
Reproducibility	0.2 A

Where A is the average result in % m/m.

No bias can be assigned since the heptane insoluble asphaltenes are defined by this test method.

BASE NUMBER

GENERAL

New and used petroleum products may contain basic constituents that are present as additives. The relative amounts of these materials can be determined by titrating with acids. The neutralization number expressed as base number, is a measure of this amount of basic substance in the oil always under the conditions of the test. The neutralization number is used as a guide in the quality control of lubricating oil formulations. It is also sometimes used as a measure of lubricant degradation in service; however, any condemning limits must be empirically established.

There are four different ASTM test methods for the determination of base numbers: D 664, D 974, D 2896, and D 4739. Test Method D 664 has always given poor precision. Hence, ASTM has cancelled that base number part of Test Method D 664, and replaced it with Test Method D 4739. The results obtained with the latter method, however, are not necessarily identical to those obtained with Test Methods D 664 or D 2896. It needs to be emphasized that:

(1) Different base number methods may give different results for the same sample.
(2) The test method should not be arbitrarily changed, since the new values may not be compatible with the historical data.
(3) Test Method D 2896 remains the preferred base number method due to its superior precision, definitive end point and large database.

See Table 4 for comparison of four base number methods.

TABLE 4—*Comparison of ASTM Test Methods for Base Number Determination.*

Area	D 664	D 974	D 2896	D 4739
Significance	Determines organic and inorganic bases, amino compounds, salts of weak acids (soaps), basic salts of polyacidic bases, and heavy metal salts.	Same as in D 664.	Same as in D 664.	Same as in D 664.
Procedure	Sample dissolved in toluene+IPA and titrated with alcoholic HCl. Uses potentiometric titrator. Slow equilibration. Titrant addition variable. Influence or buffer end point.	Sample dissolved in toluene+IPA and titrated with alcoholic HCl. Uses *p*-Naphtholbenzein as color indicator.	Sample dissolved in chlorobenzene and glacial acetic acid, and titrated with $HClO_4$ in glacial acetic acid. Uses potentiometric titrator. Fast equilibration. Continuous titrant addition. Inflection end point.	Sample dissolved in IPA+ $CHCl_3$+toluene+5 % water and titrated with alcoholic HCl. Uses potentiometric titrator. Slow equilibration. Titrant addition fixed. Inflection or buffer end point.
Scope	Applicable to fresh oils, but definitely not for used oils.	Applicable to fresh and used oils.	Applicable to fresh and used oils.	Applicable to fresh and used oils.
Limitations	Poor repeatability and reproducibility. Unsatisfactory end point. Results vary with the sample weight. Solvent does not hold all sample in solution. Highly dependent on sample's water content (±0.5 % water changes results by 100 %). Low results with overbased oils, and weak bases are not completely titrated.	Not suitable for measuring basic constituents of many basic additive type lube oils. D 664 should be used for this. Many cutting oils, rust-proofing oils, and similar compounded oils or excessively dark colored oils that cannot be analyzed by this method due to obscurity of color indicator end point can be analyzed by D 664.	With strongly overbased oil additives and nitrogenous polymeric compounds, higher results may be obtained.	Chloroform is a suspected carcinogen. Method not tested with base numbers over 70.
Advantages			Better precision. Always gives good titration breaks. Results are not dependent on sample weight. Gives good agreement with calculated results even with overbased oils. Complete titration of weak bases is obtained. Faster than D 664.	Better precision. Well defined inflection point. Good sample dissolution.
General	Results may or may not be numerically the same as those by D 974, but they are generally of the same order of magnitude.		Results similar to D 664 for many materials, but usually higher. No constant correlation between D 664 and D 2896 for all products; but consistent for same product.	Results similar to D 664 for many oils; but usually higher. No consistent correlation between D 664, D 2896 or D 4739; but consistent for same product.

BASE NUMBER BY COLOR INDICATOR TITRATION: D 974

(Equivalent Test Methods: IP 139, ISO 6618, DIN 51558T1, JIS K 2501, and AFNOR T60-112)

EXPLANATION

See discussion under Acid Number for Test Method D 974. Although this test has been used for base number of petroleum products for many decades, it may not be truly suitable for this measurement of many basic additive type lubricating oils. Test Method D 4739 can be used for this purpose. The values obtained by this test may not be numerically the same as those obtained by Test Method D 4739, but they are generally of the same order of magnitude.

TEST SUMMARY

The sample is dissolved in toluene, isopropyl alcohol containing a small amount of water and is titrated at room temperature with standard alcoholic acid solution to the end point indicated by the color change of the added *p*-naphtholbenzein solution (orange in acid and green-brown in base).

TEST PRECISION

Base Number	Repeatability	Reproducibility
0.00 to 0.1	0.03	0.04
>0.1 to 0.5	0.05	0.08
>0.5 to 1.0	0.08	
>1.0 to 2.0	0.12	
>0.5 to 2.0		15% of mean

These precision statements do not apply to highly colored oils which obscure the end point of color change. These estimates are based on the use of manual burets; precision using automated burets is not known.

The procedure has no known bias.

BASE NUMBER IN LUBRICANTS BY COLOR INDICATOR TITRATION: D 5984

EXPLANATION

This is a semi-quantitative field test method used on new and used lubricating oils. These samples can contain base constituents present as additives or as degradation products formed during service. A decrease in the measured base number is often used as a measure of lubricant degradation. This test method uses reagents that are considered less hazardous than those used in other alternate base number methods. It uses pre-packaged reagents for field use where laboratory equipment is unavailable and quick results are at a premium.

This test method covers base numbers in the range 0 to 20. Higher base numbers can be determined by diluting the sample or using a smaller sample size. Results obtained by this test method are similar to those obtained by Test Method D 2896. No general relationship between bearing corrosion and base number is known.

TEST SUMMARY

The sample is dissolved in *iso*-octane and alcoholic hydrochloric acid. The solution is mixed with NaCl solution and the aqueous and organic phases are allowed to separate. The aqueous phase is then decanted off and titrated with NaOH solution using methyl red indicator. When the solution color changes from magenta to yellow, the base number is read off the side of the titrating buret.

TEST PRECISION

Repeatability:	0.09 base number units
Reproducibility:	1.91 base number units

This test method has no bias.

BASE NUMBER POTENTIOMETRIC PERCHLORIC ACID TITRATION:

D 2896

(Equivalent Test Methods: IP 276, ISO 3771, and JIS K 2501)

For many materials the results obtained by this test method will be similar to those obtained by Test Method D 664. With certain compounds such as strongly over-based oil additives and nitrogenous polymeric compounds, higher results may be obtained. The consitutents that may be considered to have basic characteristics include organic and inorganic bases, amino compounds, salts of weak acids (soaps), basic salts of polyacidic bases, and salts of heavy metals. This test method is applicable to both fresh oils and used oils.

TEST SUMMARY

The sample is dissolved in an essentially anhydrous mixture of chlorobenzene and glacial acetic acid and titrated with a solution of perchloric acid in glacial acetic acid with a potentiometric titrimeter. A glass indicating electrode and a calomel reference electrode are used, the latter being connected with the sample solution by means of a salt bridge. The meter readings are plotted against the respective volumes of titrating solution, and the end point is taken at the inflection in the resulting curve.

Occasionally certain used oils give no inflection in the standard titration, in which case a back titration modification with sodium acetate titrant is employed.

TEST PRECISION

	Procedure	Repeatability, Percent	Reproducibility, Percent
Oils with Forward	A	3	7
Titration	B	5	7
Used Oil with Back Titration	A	24	32

NOTES ABOUT BASE NUMBER TEST

There has been concern around the world due to the use of the halogenated solvent used in this test. Attempts have been made to replace chloroform or chlorobenzene with trichloroethane, toluene, xylene, DMSO, and other solvents. To date, the results have been mixed. However, it is to be expected that some time in the future, ASTM will drop the use of halogenated solvent from this test. In one case procedure A was used with mixed xylenes instead of chlorobenzene, and alternate titration solvent (1:2 glacial acetic acid and mixed xylenes). This resulted in a repeatability of 6.2 %, and reproducibility of 16.2 %, both cases worse than in original D 2896. Addition of ~10 % acetone to alternative titration solvent reduces electrode noise. However, precision of this modification is not known.

BASE NUMBER BY POTENTIOMETRIC HCl TITRATION:

D 4739

(Equivalent Test Methods: IP 417, ISO 6619, and JIS K 2501)

EXPLANATION

Test Method D 4739 was developed as an alternative for the base number portion of Test Method D 664. Base numbers obtained by this test method may or may not be numerically the same as those obtained by the base number portion of Test Method D 664.

TEST SUMMARY

The sample is dissolved in a mixture of toluene, isopropyl alcohol, chloroform, and a small amount of water and titrated potentiometrically with alcoholic hydrochloric acid solution. An end point is selected from a titration curve and used to calculate a base number.

A color indicator titration method is also available in the Test

Method D 974. The base numbers obtained by the potentiometric method may or may not be numerically the same as those obtained by Test Method D 974. Potentiometric methods for base number are also available in Test Method D 2896.

TEST PRECISION

Repeatability:	10.4 % of mean of two results
Reproducibility:	21.1 % of mean of two results

BENZENE/TOLUENE IN GASOLINE BY GAS CHROMATOGRAPHY:

D 3606

(Equivalent Test IP 425)

EXPLANATION

Benzene is considered as a toxic material and the knowledge of its concentration can be an aid in evaluating the possible health hazards to persons handling and using the gasoline. This test method determines benzene and toluene in finished motor and aviation gasoline by gas chromatography. The range of determination includes benzene in 0.1 to 5 volume % and toluene in 2 to 20 volume % levels. The method has been tested in conventional as well as oxygenates containing gasolines. The method is not suitable for gasolines containing ethanol and methanol.

TEST SUMMARY

The sample is doped with an internal standard methyl ethyl ketone (MEK) and is then injected into a gas chromatograph equipped with two columns connected in series. The first column is packed with a nonpolar phase such as methyl silicone which separates the components according to their boiling points. The second column is packed with a highly polar phase such as 1,2,3-tris(cyanoethoxy) propane, which separates the aromatic and nonaromatic compounds. The eluted components are detected by a thermal conductivity detector and recorded on a strip chart. From the measured peak areas the concentration of each component is calculated with reference to the internal standard.

TEST PRECISION

Component	Range, Volume Percent	Repeatability	Reproducibility
Benzene	0.1–1.5	$0.03(X)+0.01$	$0.13(X)+0.05$
Benzene	>1.5	0.03	$0.28(X)$
Toluene	1.7–9	$0.03(X)+0.02$	$0.12(X)+0.07$
Toluene	>9	0.62	1.15

Where X is the mean volume % of the component.

The bias of this test method is not known.

BENZENE/TOLUENE IN GASOLINE BY INFRARED (IR) SPECTROSCOPY:

D 4053

(Equivalent Test Method: IP 429)

EXPLANATION

See the explanation in D 3606.

Toluene and heavier aromatic compounds have some interference in this test method. To minimize this interference, the procedure includes a correction. Errors due to other sources of interference may be partially compensated for by calibrating with gasoline stocks containing little or no benzene but which otherwise are similar in aromatic content to the sample to be analyzed.

TEST SUMMARY

A gasoline sample is examined by infrared spectroscopy and following a correction for interference is compared with calibration blends of known benzene concentration.

TEST PRECISION

Repeatability:	0.08 volume %
Reproducibility:	0.18 volume %

The bias of this test method has not been established.

BENZENE/TOLUENE IN ENGINE FUELS USING MID-IR SPECTROSCOPY:
D 6277

EXPLANATION

Benezene is a toxic chemical whose concentration in products is limited by government agencies. This test method is fast, simple to run, and inexpensive. It is applicable for quality control in the production and distribution of spark ignition engine fuels in the concentration range 0.1 to 5 volume %.

TEST SUMMARY

A beam of infrared light is imaged through a liquid sample cell onto a detector, and the detector response is determined. Wavelengths of the spectrum that correlate highly with benzene or interferences are selected for analysis using selective bandpass filters or mathematically by selecting areas of the whole spectrum. A multivariate mathematical analysis converts the detector response for the selected areas of the spectrum of an unknown to a concentration of benzene.

Toluene and other monosubstituted aromatics interfere. Oxygenates can interfere with measurements made with filter apparatus. Proper choice of the apparatus, proper design of a calibration matrix, and proper utilization of multivariate calibration techniques can minimize these interferences.

TEST PRECISION

Conditions	Repeatability	Reproducibility
Filter Based Mid-IR Instruments	$0.21+0.207\ X$	$0.121+0.012\ X$
FTIR Instruments with PLS Calibration Instruments	$0.013+0.052\ X$	$0.022+0.118\ X$
FTIR Instruments using Classical Least Squares Calibration	$0.047+0.043\ X$	$0.099+0.031\ X$

Where X is the benzene concentration determined.

Bias—Bias against reference materials is not known. However, there appears to be a variable bias against Test Method D 5769, of the order of −0.06 vol. % for the FTIR procedure and +0.06 vol. % for the filter procedure.

AEROBIC, AQUATIC BIODEGRADABILITY OF LUBRICANTS IN A CLOSED RESPIROMETER:
D 6731

EXPLANATION

This test method covers a procedure for determining the degree of biodegradability of lubricants or their components in an aerobic aqueous medium on exposure to an inoculum under controlled laboratory conditions. The test method is an ultimate biodegradation test that measures oxygen demand in a closed respirometer. This test method is suitable for evaluating the biodegradability of volatile as well as non-volatile lubricants or lubricant components. This test method is applicable to lubricants and lubricant components which are not toxic and not inhibitory to the test microorganisms at the test concentration.

Because of the stringency of this test method, low results do not necessarily mean that the test material is not biodegradable under environmental conditions, but indicate that further testing is necessary to establish biodegradability. The water solubility or dispersibility of the lubricant or component may influence the results obtained and hence comparison of test results may be limited to lubricants or components with similar solubilities. The behaviors of complex mixtures are not always consistent with the individual properties of the components.

TEST METHOD SUMMARY

Biodegradability is determined by measuring the oxygen consumed when the test material is exposed to microorganisms under controlled aerobic aquatic conditions. This value is then compared to the theoretical amount of oxygen which is required to oxidize all the elements (i.e., carbon, hydrogen, nitrogen, and so forth) in the test material. The test material is mixed with aerobic microorganisms in a closed respirometer containing a defined aquatic medium and measures the biodegradation of the test material by following the decrease in oxygen in the respirometer.

TEST PRECISION

The precision and bias of this test method is not determined as of yet.

BLOCKING AND PICKING POINTS OF PETROLEUM WAX:

D 1465

EXPLANATION

Blocking of waxed paper, because of the relatively low temperature at which it may occur, can be a major problem to the paper coating industry. The wax picking and blocking points indicate an approximate temperature range at or above which waxed surfaces in contact with each other are likely to cause surface film injury.

The wax blocking point is the lowest temperature at which film disruption occurs across 50 % of the waxed paper surface when the test strips are separated.

TEST SUMMARY

Paper test specimens are coated with the wax sample, folded with the waxed surfaces together, and placed on a blocking plate which is heated at one end and cooled at the other to impose a measured temperature gradient along its length. After a conditioning period on the plate, the specimens are removed, unfolded, and examined for film disruption. The temperatures of corresponding points on the blocking plate are reported as the picking and blocking points or as blocking range.

TEST PRECISION

Analysis	Repeatability	Reproducibility
Picking Point	2.8°C(5°F)	3.6°C(6.5°F)
Blocking Point	1.7°C(3°F)	3.3°C(6°F)

This test method has no bias.

BOILING RANGE DISTRIBUTION OF CRUDE PETROLEUM BY GAS CHROMATOGRAPHY:

D 5307

EXPLANATION

Boiling range distribution is an essential requirement of crude oil assay. This information can be used to refinery yields, and to evaluate the economics of using one particular crude as opposed to another. The method covers boiling range distribution of water-free crude petroleum through 538°C(100°F). Material boiling above this temperature is reported as residue. The method is applicable to whole crude samples that can be dissolved in a solvent to permit sampling using a microsyringe.

This test method is faster than Test Method D 2892 and can be used when only small volumes of sample are available. Also, this test method gives results up to 538°C while Test Method D 2892 is limited up to 400°C. Results by both test methods are equivalent.

TEST SUMMARY

A solution of crude oil in carbon tetrachloride is injected into a gas chromatographic column that separates hydrocarbons by their boiling point order. The column temperature is raised at a reproducible and linear rate. The area under the chromatogram is recorded throughout the run. Calibration is done using a mixture of *n*-paraffins of known boiling point through 538°C. The amount of sample boiling above this range is estimated by a second analysis in which an internal standard is added to the crude oil.

TEST PRECISION[1]

Repeatability and Reproducibility.

Percent Off	Repeatability, °C	Reproducibility, °C
IBP	3.7	10.6
5	4.7	14.8
10	6.9	11.3
20	6.8	15.4
30	7.6	20.4
40	9.3	24.6
50	10.6	30.3
60	11.8	25.9
70	17.6	39.2
80	24.8	38.8
85	18.8	38.8
90	20.7	44.9
Residue	2.6 Mass %	8.1 Mass %

[1] This table is Table 3 from Test Method D 5307.

BOILING RANGE DISTRIBUTION OF GASOLINE BY WIDE-BORE CAPILLARY GAS CHROMATOGRAPHY: D 7096

EXPLANATION

The determination of the boiling range distribution of gasoline by gas chromatographic simulated distillation (CG-SD) provides an insight into the composition of the components from which the gasoline was blended. This knowledge is useful for the control of refinery processes and for the blending of finished gasoline. This method has better precision than the conventional distillation by Test Method D 86. Additionally, this test method provides more accurate and detailed information about the composition of the light ends. The distillation data produced by this test method are similar to that which would be obtained from a cryogenic, true boiling point (15 theoretical plates) distillation. The method is applicable to petroleum products and fractions with a final boiling point of 280°C or lower, as measured by this test method. This test method is designed to measure the entire boiling range of gasoline and gasoline components with either high or low vapor pressure and is commonly referred to as Simulated Distillation (SimDis) by gas chromatographers. This test method has been validated for gasolines containing ethanol. Gasolines containing other oxygenates are not specifically excluded, but they were not used in the development of this test method. This test method can estimate the concentration of n-pentane and lighter saturated hydrocarbons in gasoline.

TEST METHOD SUMMARY

The sample is vaporized and transported by carrier gas into a nonpolar, wide-bore capillary gas chromatographic column. The column temperature is raised at a reproducible, linear rate so as to elute the hydrocarbon components in boiling point order for measurement by a flame ionization detector. Conditions are selected such that n-pentane and lighter saturated hydrocarbons in the calibration mixture are resolved discretely. Linear correlation between hydrocarbons boiling point and retention time is established using a known mixture of hydrocarbons covering the boiling range expected in the sample. Area slices are converted to volume using theoretical hydrocarbon volume response factors. Oxygenated samples require experimental determinations of oxygenate response factors.

INTERFERENCES—Ethanol or other oxygenates may coelute with hydrocarbons present in the sample. Since the response of the oxygenates is substantially different from the response of the hydrocarbons, response factors are used to correct the area slice for the elution interval of oxygenates. Concentration of n-pentane and lighter saturated components may be estimated from the analysis. However, early eluting olefins present in the gasoline samples may coelute with these compounds.

TEST PRECISION

The currently available precision data are based on limited interlaboratory test results. Further data will be developed in the future. Based on the results from three laboratories, analyzing the same sample, following repeatability standard deviation was obtained:

	°C		°C		°C
IBP	1.3	2 %	1.3	5 %	1.3
10 %	1.4	20 %	1.3	30 %	1.2
40 %	1.3	50 %	1.3	60 %	1.5
70 %	1.7	75 %	1.9	80 %	1.7
85 %	1.7	90 %	1.8	95 %	2.4
98 %	3.2	FBP	4.3		

For gases, the repeatability values are as follows:

Hydrocarbon	C_3	i-C_4	n-C_4	i-C_5
Vol % Gas	0.05	0.04	0.15	0.20

The reproducibility of this test method is being determined. The bias in results of this test method cannot be determined because the boiling range distribution is defined by this test method.

BOILING RANGE DISTRIBUTION OF PETROLEUM DISTILLATES BY GAS CHROMATOGRAPHY:
D 7213

EXPLANATION

The boiling range distribution of light and medium petroleum distillate fractions provide an insight into the composition of feed stocks and products related to the petroleum refining process. This gas chromatographic (GC) method can be used to replace the conventional distillation methods. This test method extends the scope of boiling range determination by GC to include light and medium petroleum distillate fractions beyond the scope of Test Method D 2887 and below Test Method D 6352. The boiling range distributions obtained by this Test Method are theoretically equivalent to those obtained by true boiling point distillation (D 2892). They are, however, not equivalent to results obtained from low efficiency distillation such as Test Method D 86 or D 1160.

This method is applicable to petroleum distillates having an initial boiling point greater than 100°C and a final boiling point less than 615°C at atmospheric pressure. This method is not applicable to petroleum distillates containing low molecular weight components such as napthas, reformats, gasolines, crude oils. Materials containing heterogeneous components, for example, alcohols, ethers, acids, or esters, or residue are not to be analyzed by this method.

TEST SUMMARY

The boiling range distribution by distillation is simulated by the use of gas chromatography. The solvent should not

TABLE 5—*Repeatability*[a]

% OFF	°C	°F
IBP	3.61	6.45
5	2.38	4.29
10	2.58	4.65
20	2.81	5.07
30	3.15	5.68
40	3.24	5.83
50	3.36	6.05
60	3.29	6.05
70	3.20	5.76
80	2.96	5.34
90	2.76	4.98
95	2.96	5.32
97	3.31	5.95
99	4.91	8.84
FBP	6.27	11.28

[a] The precision statement was derived from a robustness study conducted in 2004 using four samples and four laboratories. An interlaboratory study in accordance with D 6300 is underway to estimate permanent precision for this test method.

interfere with measurement of the samples in the 100 to 615°C range, and it should be apolar. A non-polar open tubular capillary GC column is used to elute the hydrocarbon components of the sample in order of increasing boiling point.

A sample aliquot is diluted with a viscosity reducing solvent and introduced into the GC system. Sample vaporization is provided by separate heating of the point of injection or in conjunction with column oven heating. The column oven temperature is raised at a reproducible linear rate to effect separation of the hydrocarbon components in order of increasing boiling point. The elution of sample components is quantitatively determined using a flame ionization detector. The detector signal integral is recorded as area slices for consecutive retention time intervals during the analysis. Retention times of known normal paraffin hydrocarbons spanning the scope of this test method (C_5 through C_{60}) are determined and correlated to their boiling point temperatures. The normalized cumulative corrected sample areas for each consecutive recorded time interval are used to calculate the boiling range distribution. The boiling point temperature at each reported percent off increment is calculated from the retention time calibration.

TEST PRECISION

Repeatability of this method based on a preliminary robustness study is as follows. Reproducibility of the method is not known at present. No bias of this method can be determined because the boiling point distribution can be defined only in terms of a test method.

BOILING RANGE DISTRIBUTION OF GASOLINE FRACTIONS BY GAS CHROMATOGRAPHY:

D 3710

EXPLANATION

The determination of the boiling range distribution of gasoline by GC distillation helps understand the composition of the components from which the gasoline was blended. It facilitates on-line controls at the refinery and its results offer improved means of describing several car performance parameters. This test method covers petroleum products and fractions with a final boiling point of 500°F(260°C) or lower as measured by this test method. This test method is designed to measure the entire boiling range of gasoline with either high or low Reid vapor pressure.

TEST SUMMARY

The sample is injected into a gas chromatographic column which separates hydrocarbons in boiling point order. The column temperature is raised at a reproducible rate and the area under the chromatogram is recorded throughout the run. The calibration is done using a known mixture of hydrocarbons covering the boiling range expected in the sample.

TEST PRECISION[1]

The precision of this test method depends upon the shape of the boiling range distribution curve. Precision varies with the percent recovered and the rate of temperature change with percent recovered: dT/dV, where T is the temperature and V is the percent recovered.

Bias of this test method is not known.

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[1] These tables are part of Table 1 and Table 2 from Test Method D 3710.

Repeatability as a Function of Percent Recovered and dT/dV.

	Repeatability, r, °F (°C)										
Volume Percent Recovered, dT/dV:	0	2(1)	4(2)	6(3)	8(4)	10(6)	12(7)	14(8)	20(11)	30(17)	40(22)
IBP	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)
1	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)	—	—	—
5	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)	—	—	—
10	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)	2(1)	—	—	—
20	2(1)	3(2)	4(2)	5(3)	7(4)	10(6)	—	—	—	—	—
30 to 90	2(1)	3(2)	4(2)	5(3)	7(4)	10(6)	14(8)	19(10)	—	—	—
95	—[a]	—	2(1)	3(2)	5(3)	7(4)	9(6)	13(7)	—	—	—
99	—	—	—	—	—	—	—	6(3)	6(3)	6(3)	6(3)
FBP	—	—	—	—	—	—	—	—	6(3)	6(3)	6(3)

[a] (—) Outside the range observed in the cooperative study.

Reproducibility as a Function of Percent Recovered and dT/dV.

	Reproducibility, R, °F (°C)[a]										
Volume Percent Recovered, dT/dV:	0	2(1)	4(2)	6(3)	8(4)	10(6)	12(7)	14(8)	20(11)	30(17)	40(22)
IBP	7(4)	7(4)	8(4)	8(4)	8(4)	8(4)	8(4)	8(4)	9(5)	10(6)	12(7)
1	5(3)	5(3)	5(3)	6(3)	6(3)	6(3)	6(3)	6(3)	—	—	—
5	5(3)	5(3)	5(3)	5(3)	5(3)	5(3)	5(3)	6(3)	—	—	—
10	6(3)	6(3)	6(3)	6(3)	6(3)	6(3)	3(7)	—	—	—	—
20	5(3)	7(4)	11(6)	16(9)	23(13)	30(17)	—	—	—	—	—
30 to 90	6(3)	9(5)	13(7)	20(11)	27(15)	36(20)	46(26)	55(31)	—	—	—
95	—[b]	—	11(6)	16(9)	23(13)	30(17)	38(21)	46(26)	—	—	—
99	—	—	—	—	—	—	—	20(11)	24(13)	33(18)	36(20)
FBP	—	—	—	—	—	—	—	—	19(11)	26(14)	36(20)

[a] For thermal conductivity detectors. For flame ionization detectors (FID) reproducibilities, R, are the same except in the 20 to 95 % recovered range where: $R_{FID} = 9.90 R_{TCD}$.

[b] (—) Outside the range observed in the cooperative study.

BOILING RANGE DISTRIBUTION OF PETROLEUM DISTILLATES BY GAS CHROMATOGRAPHY: **D 6352**

EXPLANATION

The boiling range distribution of medium and heavy petroleum distillate fractions provides an insight into the composition of feed stocks and products related to the petroleum refining process. The gas chromatographic (GC) simulation of this determination is used to replace conventional distillation methods for control of refining operations. This method can be used for product specification testing with the mutual agreement of interested parties. The test method is applicable to petroleum distillate fractions with an initial boiling point of <700°C at atmospheric pressure. The test method is not applicable to products containing low molecular weight components, for example, napthas, reformates, gasolines, and crude oils. Do not use this test method for materials containing heterogenous components (for example, alcohols, ethers, esters, or acids) or residue.

This test method extends the scope of Test Method D 2887 to boiling range determination by GC to include medium and heavy petroleum distillate fractions. No correlations have been established between the boiling range distributions obtained by this test method and those obtained by using Test Methods D 86 or D 1160.

TEST SUMMARY

A nonpolar open tubular capillary GC column is used to elute the hydrocarbon components of the sample in order of increasing boiling point. A sample aliquot diluted with a viscosity reducing solvent is introduced into the chromatographic system. The column oven temperature is raised at a specified linear rate to effect separation of the hydrocarbon components. The detector signal is recorded as area slices for consecutive retention time intervals during the analysis. Retention times of known normal paraffin hydrocarbons spanning the scope of the test method are used for normalizing the retention times of the unknown mixture area slices.

TEST PRECISION[1]

See the table below for the test precision.

No statement of bias can be made for this test method.

Repeatability and Reproducibility of Temperature as a Function of Percent Recovered Using a 100 % Dimethylpolysiloxane Stationary Phase Column.

Mass % Recovered	Repeatability, °C	Repeatability, (°F)	Reproducibility, °C	Reproducibility, (°F)
0.5 (IBP)	8.1	(14.6)	49.1	(88.4)
2	3.7	(6.7)	15.4	(27.7)
5	2.3	(4.1)	9.0	(16.2)
10	2.8	(5.0)	7.1	(12.8)
20	2.7	(4.9)	6.2	(11.2)
30	2.4	(4.3)	5.9	(10.6)
40	2.6	(4.7)	6.0	(10.8)
50	2.7	(4.9)	6.4	(11.5)
60	2.4	(4.3)	6.4	(11.5)
70	3.0	(5.4)	7.2	(13.0)
80	3.0	(5.4)	7.8	(14.0)
90	3.4	(6.1)	10.5	(18.9)
95	4.7	(8.5)	14.3	(25.7)
98	6.3	(11.3)	21.8	(39.2)
99.5 (FBP)	13.9	(25.0)	38.1	(68.6)

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[1] This table is Table 7 from Test Method D 6352.

BOILING POINT DISTRIBUTION OF CRUDE OILS BY HIGH TEMPERATURE GAS CHROMATOGRAPHY: D 7169

EXPLANATION

The determination of boiling point distribution of crude oils and vacuum residues, as well as other petroleum fractions provides important information for refinery operations as to the potential mass percent yield of products. This can aid in establishing operational conditions in the refinery. The amount of residue (or sample recovery) is determined using an external standard. The knowledge of such amount of residue produced is important in determining the economics of the refining process.

This test method extends the applicability of simulated distillation to samples that do not elute completely from the chromatographic system. This test method is used to determine the boiling point distribution through a temperature of 720°C which corresponds to the elution of n-C_{100}. The method is used for analysis of crude oils and atmospheric and vacuum residues using capillary columns with thin films which result in the incomplete separation of C_4–C_8 in the presence of large amounts of carbon disulfide, and thus yields an unreliable boiling point distribution corresponding to this elution interval. Additionally, quenching of the response of the detector employed to hydrocarbons eluting during carbon disulfide elution, results in unreliable quantitative analysis of the boiling distribution in the C_4–C_8 region. Since the detector does not quantitatively measure the carbon disulfide, its subtraction from the sample using a solvent-only injection and corrections to this region via quenching factors, results in an approximate determination of the net chromatographic area. A separate high resolution gas chromatographic analysis of the light end portion of the sample may be necessary in order to obtain a more accurate description of the boiling point curve in the interval in question.

This method is not applicable to the analysis of materials containing a heterogenous component such as polyesters and polyolefins.

TEST SUMMARY

This gas chromatographic method utilizes an inlet and a capillary column, both of which are subject to a temperature program. A flame ionization detector is used as a transducer that converts mass to an electrical signal. A data acquisition system operating in the slice mode and chromatography software is used to accumulate the electronic signal. A retention time calibration mixture is used to develop a retention time versus boiling point curve. A solution of the Reference Oil 5010, which fully elutes from the column under the test conditions, and whose boiling point distribution has

TABLE 6—*Repeatability Std. Dev. of the Boiling Point Distribution.*

	Crude Oil Sample		Residue Sample	
% Off	°C	°F	°C	°F
IBP	0.55	1	2.8	2.8
5	1.4	2.6	1.9	1.9
10	1.3	2.3	1.8	1.8
15	1.9	3.4	1.6	1.6
20	2.6	4.7	1.7	1.7
25	2.9	5.4	1.8	1.8
30	3.0	5.4	1.9	1.9
35	3.3	5.9	2.1	2.1
40	3.5	6.4	2.5	2.5
45	4.3	7.7	2.7	2.7
50	4.7	8.5	3.2	3.2
55	5.3	9.6	3.8	3.8
60	6.4	11.5	3.7	3.7
65	7.2	13	4.8	4.8
70	8.6	15.6	6.3	11.3
75	9.4	17	6.1	10.9
80	11.6	21	7.4	13.3
85	16.7	30	8.6	15.4
90	19.4	35	9.4	17

TABLE 7—*Repeatability Std. Dev. of % Mass in Selected Cut Point Intervals.*

Outpoints Intervals °C (°F)	Repeatability for Crude Oils Mass %	Repeatability for Residues Mass %
27–165 (80–329)	0.65	…
165–220 (329–428)	0.31	…
220–330 (428–626)	0.62	0.22
330–450 (626–842)	0.82	0.59
450–600 (842–1112)	1.17	1.51
600–720 (1112–1328)	0.99	1.31
Recovery	2.77	3.01

been characterized by Test Method D 6352, is used to determine the detector response factor. Solvent injections are made, and the resulting signal is subtracted from both the response factor standard and the sample chromatogram.

Finally, the sample solution is injected, and with the use of the response factor, the amount of sample recovered is calculated. After converting the retention times of the sample slices to temperature, the boiling point distribution can be calculated up to the recovered amount.

TEST PRECISION

A preliminary repeatability of the test method was determined employing a limited round robin. The data are given in the following Tables 6 and 7. The reproducibility of the method is not available at present.

No information on bias is available because there is no accepted reference material for such analysis.

BORDERLINE PUMPING TEMPERATURE OF ENGINE OILS:
D 3829

EXPLANATION

Borderline pumping temperature is a measure of the lowest temperature at which an engine oil can be continuously and adequately supplied to the oil pump inlet of an automotive engine. This test method covers the prediction of the borderline pumping temperature (BPT) of engine oils through the use of a 16-h cooling cycle over the temperature range from 0 to −40°C. Applicability to petroleum products other than engine oils has not been determined.

TEST SUMMARY

An engine oil sample is cooled from 80°C to the desired test temperature at a nonlinear programmed cooling rate over a 10-h period and held at the test temperature for the remainder of a 16-h period. After completion of the soak period, two standard torques of increasing severity are applied to the rotor shaft and the speed of rotation in each case is measured. From the results at three or more temperatures, the borderline pumping temperature is determined.

Alternatively, for some specification or classification purposes it may be sufficient to determine that the BPT is less than a certain specified temperature.

TEST PRECISION

Repeatability:	1.3°C
Reproducibility:	3.2°C

BROMINE NUMBER OF DISTILLATES AND ALIPHATIC OLEFINS:
D 1159
(Equivalent Test Methods: IP 130 and ISO 3839)

EXPLANATION

Bromine Number is the grams of bromine that will react with 100 g of the sample under the test conditions. The magnitude of bromine number is an indication of the quantity of bromine-reactive constituents, not an identification of constituents. It is used as a measure of aliphatic unsaturation in petroleum samples, and as percentage of olefins in petroleum distillates boiling up to approximately 315°C(600°F).

TEST SUMMARY

A known weight of the sample dissolved in a specified solvent maintained at 0 to 5°C(32 to 41°F) is titrated with standard bromide-bromate solution. The end point is indicated by a dead stop electrometric titration apparatus when the presence of free bromine causes a sudden change in the electrical conductivity of the system.

TEST PRECISION

	Repeatability	Reproducibility
90% Distillation Point $<$205°C	0.11 ($X^{0.7}$)	0.72 ($X^{0.7}$)
90% Distillation Point between 205 and 327°C	3	12

Where X is the sample mean.

BROMINE NUMBER BY ELECTROMETRIC TITRATION:
D 2710

EXPLANATION

This test method determines the amount of bromine reactive materials in petroleum hydrocarbons and is thus a measure of trace amounts of unsaturation in these materials. An estimate of the quantity of these materials is useful in assessing the suitability of lighter fractions for use as reaction solvents.

It is applicable to materials with bromine indexes below 1000. Materials with higher than this bromine index should be tested using Test Method D 1159. Industrial aromatic hydrocarbons should be tested using Test Method D 1492. This test method is applicable only to essentially olefin-free hydrocarbons or mixtures that are substantially free from materials that are lighter than isobutane and have a distillation end point under 288°C(550°F).

TEST SUMMARY

A sample dissolved in a specified solvent is titrated with standard bromide-bromate solution. The end point is indicated by a dead drop electrometric titration apparatus, when the presence of free bromine causes a sudden change in the electrical conductivity of the system.

TEST PRECISION

Repeatability:	14
Reproducibility:	118

This test method has no bias.

BURNING QUALITY OF KEROSENE
D 187

EXPLANATION

This test method covers the qualitative determination of the burning properties of kerosene to be used for illuminating purposes. This method is referred to in the kerosene specifications. Corresponding IP Method 10 features a quantitative evaluation of the wick-char-forming tendencies of kerosene. Both methods subject the kerosene to somewhat more severe operating conditions than would be experienced in typical designated applications.

TEST SUMMARY

A kerosene sample is burned for 16 h in a specified lamp under specified conditions. The average rate of burning, the change in the shape of the flame, and the density and color of the chimney deposit are reported.

TEST PRECISION

Measurement	Repeatability	Reproducibility
Flame Height, mm	$(36.2/X)^3$	$(42.7/X)^3$
Flame Width, mm	$(37.8/X)^3$	$(45.2/X)^3$
Burning Rate, g/h	$(26.5/X)^3$	$(36.2/X)^3$
Chimney Deposit Color	any difference	one category
Chimney Deposit Density	any difference	one category

Where X is the average of two values.
The bias data have not yet been collected.

BUTYLENE ANALYSIS BY GC: D 4424

EXPLANATION

This test method can be used to determine butylenes stream composition for custody transfer payments. It can also provide data necessary to evaluate processing requirements in an operating plant. The method covers the gas chromatographic analysis of commercial butylenes, butylenes concentrates, and butane-butylene mixtures. It does not cover high purity butene-1 or high purity isobutene streams, or both. However, it is possible that one or more columns listed in the method may be capable of the separation necessary for high-purity analyses.

This test method covers the following components at about 0.05 % or greater: propane, propylene, isobutene, n-butene, isobutene, trans-butene-2, cis-butene-2, 1,3-butadiene, isopentane, and n-pentane. It is not intended for trace hydrocarbon analysis.

TEST SUMMARY

The sample is separated in a gas chromatograph system using a packed chromatographic column with either helium or hydrogen as the carrier gas. The separated components of the sample are detected either by a thermal conductivity detector or by a flame ionization detector. Calibration data are obtained by using either relative response factors or by using a standard calibration blend.

TEST PRECISION

The test method precision has not yet been developed. Bias cannot be determined since no accepted standard reference material is available for this method.

CARBON NUMBER DISTRIBUTION: D 2887
(Equivalent Test Methods: IP 321, ISO 3924, and JIS K 2254)

EXPLANATION

The determination of the boiling range distribution of petroleum fractions by gas chromatography is a rapid analytical tool, which may be used to replace conventional distillation methods for control of refining operations and specification testing. Data derived by this test method are essentially equivalent to that obtained by TBP distillation (Test Method D 2892). They are not equivalent to results from low efficiency distillations such as those obtained with Test Methods D 86 or D 1160. This test method is applicable to petroleum products and fractions with a final boiling point of 538°C(1000°F) or lower at atmospheric pressure as measured by this test method. This test method is not designed for use with gasoline samples or gasoline components. These samples must be analyzed by Test Method D 3710. This test method is limited to samples having a boiling range greater than 55°C(100°F), and having a vapor pressure sufficiently low to permit sampling at ambient temperature.

A correlation of 17 % oil volatility by GC-SD methods (Test Methods D 2887 or D 5480) has been found equivalent to 21.5 % volatility by Noack method.

DEFINITIONS

Initial Boiling Point—the point at which a cumulative area count equal to 0.5 % of the total area under the chromatogram is obtained.

Final Boiling Point—the point at which a cumulative area count equal to 99.5 % of the total area under the chromatogram is obtained.

TEST SUMMARY

The boiling range distribution determination by distillation is simulated by the use of gas chromatography. A nonpolar packed or open tubular (capillary) gas chromatographic column is used to elute the hydrocarbon components of the sample in order of increasing boiling point. The column temperature is raised at a reproducible linear rate and the area under the chromatogram is recorded throughout the analysis. Boiling points the assigned to the time axis from a calibration curve obtained under the same chromatographic conditions by analyzing a known mixture of hydrocarbons covering the boiling range expected in the sample. From these data, the boiling range distribution can be obtained.

TEST PRECISION

Repeatability.

NOTE—x = the average of the two results in °C and y = the average of the two results in °F.

	Repeatability	
% Off	°C	°F
IBP	0.011 x	0.011 $(y-32)$
5 %	0.0032 $(x+100)$	0.0032 $(y+148)$
10–20 %	0.8	1.4
30 %	0.8	1.4
40 %	0.8	1.4
50–90 %	1.0	1.8
95 %	1.2	2.2
FBP	3.2	5.8

Reproducibility.

NOTE—x = the average of the two results in °C and y = the average of the two results in °F.

	Reproducibility	
% Off	°C	°F
IBP	0.066 x	0.06 $(y-32)$
5 %	0.015 $(x+100)$	0.015 $(y+148)$
10–20 %	0.015 $(x+100)$	0.015 $(y+148)$
30 %	0.013 $(x+100)$	0.013 $(y+148)$
40 %	4.3	7.7
50–90 %	4.3	7.7
95 %	5.0	9.0
FBP	11.8	21.2

CARBON RESIDUE BY MICRO METHOD: D 4530

(Equivalent Tests: IP 398 and ISO 10370)

EXPLANATION

The carbon residue values of the various petroleum materials serve as an approximation of the tendency of the material to form carbonaceous type deposits under degradation conditions similar to those used in the test method, and can be useful as a guide in the manufacture of certain stocks. The test results are equivalent to those by Conradson Carbon Method, Test Method D 189; however, this test method has the advantage of better control of test conditions, smaller samples, and less operator attention. Up to twelve samples can be run simultaneously. This test method is applicable to petroleum products that partially decompose on distillation at atmospheric pressure and was tested in the carbon residue range of 0.10 to 30 m/m %.

Ash-forming constituents as defined in Test Method D 482, or nonvolatile additives present in the sample will get included in the total carbon residue reported. Also, in diesel fuels thepresence of alkyl nitrates such as amyl, hexyl, or octyl nitrate causes a higher carbon residue value leading to the erroneous conclusion as to the coke-forming tendency of the fuel.

TEST SUMMARY

A weighed quantity of the sample placed in a glass vial is heated to 500°C under an inert (nitrogen) atmosphere in a controlled manner for a specific time. The sample undergoes coking reactions and volatiles formed are swept away by the nitrogen gas. The carbonaceous-type residue remaining is reported as a percent of the original sample as “carbon residue (micro)”.

TEST PRECISION

Repeatability:	$(\% \text{ carbon residue})^{0.66} \times 0.0770$
Reproducibility:	$(\% \text{ carbon residue})^{0.66} \times 0.2451$

This test method has no bias.

CARBONIZABLE SUBSTANCES IN PARAFFIN WAX: D 612

EXPLANATION

This test method determines the carbonizable substances in pharmaceutical paraffin wax whose quality is prescribed by the U.S. National Formulary. Such waxes have a melting point between 117 and 149°F (47 and 65°C), as determined by Test Method D 87.

TEST SUMMARY

Five mL of melted wax are treated with 5 mL of concentrated nitrogen-free sulfuric acid at 158°F(70°C). The color of the acid layer is compared with that of a colorimetric reference standard. If the color is not darker than the standard, then the wax is reported as passing the test.

TEST PRECISION

Since this is a pass-fail test, no precision or bias estimate can be made.

CARBONIZABLE SUBSTANCES IN WHITE MINERAL OIL: D 565

EXPLANATION

This test method is applicable to white mineral oil to check its conformance to standard of quality required for pharmaceutical use as defined by the U.S. Pharmacopeia and the National Formulary, or the Food and Drug Administration.

TEST SUMMARY

The mineral oil is treated with concentrated sulfuric acid under prescribed conditions and the resulting color is compared with a reference standard to determine whether it passes or fails the test. When the oil layer shows no change in color and when the acid layer is not darker than the reference standard colorimetric solution, the oil is reported as passing the test. A bluish haze or a slight pink or yellow color in the oil layer should not be interpreted as a change in color.

TEST PRECISION

This is a pass-fail test and no estimates of precision or bias can be made.

CARBONYLS IN C_4 HYDROCARBONS: D 4423

EXPLANATION

The determination of the carbonyl content of polymerization-grade 1,3-butadiene is necessary, since in some polymerization reactions, the presence of carbonyls in excess over some specified amount can have a deleterious effect upon the polymer properties or the reaction itself, or both.

This test method covers the determination of carbonyls (ketones and aldehydes) in C_4 hydrocarbons. The applicable range of this test method is 0–50 mg/kg carbonyls calculated as acetaldehyde. Other C_4 hydrocarbons and their mixtures besides polymerization grade 1,3-butadiene could be tested using this method. However, precision of such analysis is not known.

TEST METHOD SUMMARY

A measured amount of sample is added to an alcoholic hydroxylamine hydrochloride solution that has been adjusted to a given coloration using either alcoholic acid or base. The carbonyls will react with the hydroxylamine hydrochloride releasing an equivalent amount of hydrochloric acid which is then back-titrated to the original coloration. A blank containing only methanol and sample is titrated and the sample's results are calculated using the blank adjustment. Results are reported as mg/kg carbonyls as acetaldehyde.

TEST PRECISION

Repeatability	14 % of *X*
Reproducibility	88 % of *X*

Where *X* is the average of two test results.

No statement of bias is being made since no accepted reference materials for this test are available.

CARBONYL SULFIDE IN PROPYLENE BY GAS CHROMATOGRAPHY: D 5303

EXPLANATION

In processes producing propylene, COS usually remains with the C_3 hydrocarbons, and must be removed since it affects the product quality. COS acts as a poison to commercial polymerization catalysts, resulting in deactivation and costly process downtime. This test method overcomes a unique physical problem involved in COS measurement due to its reactive and absorptive nature and trace levels present. The test method permits several calibration techniques. For best agreement between two labs it is recommended that they use the same calibration technique. The method is applicable to COS concentrations from 0.5–4.0 mg/kg; however, the lower limit of detection of this method is believed to be below 0.1 mg/kg.

TEST SUMMARY

The test method gives a procedure for removing a sample from the sample cylinder, separating COS from propylene, detecting COS, calibrating the detector, quantitating COS content in the sample, and assaying the gas standard. A relatively large amount of sample is injected into a gas chromatograph having a single packed column, operated isothermally at 10–50°C, that separates COS from propylene. COS is detected with a flame photometric detector. Calibration data based on peak areas are obtained using a known gas standard blend of COS in the range expected for the sample. The COS peak area in the sample is measured and the concentration of COS calculated.

Hydrogen sulfide and sulfur dioxide can be present in the propylene and must be separated from COS. This can be done with the Carbopack BHT 100 columns or with the Chromosil 300 columns.

TEST PRECISION

Repeatability	0.15 X
Reproducibility	1.0 X

Where X is the average of two results in mg/kg.

The bias of this method has not been determined since there is no acceptable reference material suitable for this analysis.

DERIVED CETANE NUMBER OF DIESEL FUELS OILS: D 7170

EXPLANATION

The ignition delay (ID) and derived cetane number (DCN) values determined by this test method can provide a measure of the ignition characteristics of diesel fuel oil in compression ignition engines. The relationship of diesel fuel oil DCN determinations to the performance of full-scale, variable-speed, variable-load diesel engines is not completely understood. This test method is applicable to products typical of specification D 975, Grades No. 1-D and 2-D regular and low-sulfur diesel fuel oils, European standard EN 590, and Canadian standard CAN/CGSB-3.517-2000 and 3.6-2000. This test method may be applied to nonconventional fuels. This test determines ignition characteristics and requires a sample of approximately 220 mL and a test time of approximately 20 min on a fit-for-use instrument. The method measures the ignition delay and utilizes a constant volume combustion chamber with direct fuel injection into heated, compressed air. An equation converts an ID derived determination to a DCN. This test method covers the ID range from 2.90 to 4.35 ms (60.0 to 40.0 DCN). The combustion analyzer can measure shorter and longer ignition delays but precision may be affected.

TEST METHOD SUMMARY

A small specimen of diesel fuel oil is injected into a heated, temperature-controlled constant volume chamber, which has been previously charged with compressed air. Each injection produces a single-shot, compression ignition combustion cycle. IDs measured using sensors that detect the start of fuel injection and the start of significant combustion for each cycle. A complete sequence comprises two preliminary cycles and 25 further cycles. The ID measurements for the last 25 cycles are averaged to produce the D result. An equation converts the ID result to a DCN value.

INTERFERENCES—Minimize exposure of sample fuels, calibration materials, QC samples, and check standard to sunlight or fluorescent lamp UV emissions to minimize induced chemical reactions that can affect ignition delay measurements. Exposure of these fuels and materials to UV wavelengths shorter than 550 nm for a short period of time can significantly affect ignition delay measurements. The formation of peroxide and radicals can affect ID measurement. These formations are minimized when the sample or material stored in the dark in a cold room at a temperature of less than 10°C and covered by a blanket of nitrogen.

TEST PRECISION

Repeatability:	ID = 0.054	DCN = 0.71
Reproducibility:	Not Known	Not Known
Bias:	Not Known	Not Known

CHLORINE BOMB METHOD: D 808

GENERAL

The knowledge of amount of chlorine containing compounds present in petroleum products can be used to predict performance or handling characteristics of the product.

TEST SUMMARY

The sample is oxidized by combustion in a bomb containing oxygen under pressure. The chlorine compounds thus liberated are absorbed in a sodium carbonate solution and the amount of chlorine present is determined gravimetrically by precipitation as silver chloride.

TEST PRECISION

Chlorine, Weight Percent	Repeatability	Reproducibility
0.1 to 1.9	0.07	0.10
2.0 to 5.0	0.15	0.30
>5.0	3 %	5 %

CHLORINE FIELD TEST KIT METHOD FOR CHLORINE IN USED PETROLEUM PRODUCTS: D 5384

EXPLANATION

Chlorinated compounds can corrode the equipment and poison the refining catalysts. They are also a health hazard when incompletely combusted. Federal regulations mandate that often the chlorine content of used oil must be determined before recycling. This method can be used to determine when a used petroleum product meets or exceeds requirements for total halogen measured as chloride. It is specifically designed for used oils permitting on-site testing at remote locations by nontechnical personnel. Bromide and iodide are also titrated and reported on a molar basis as chlorine. A positive bias will result from samples that contain greater than 3 m/m % total sulfur. Other analytical methods should be used on oils containing high amounts of sulfur.

Method A provides greater or less than estimate of 1000 ppm total chlorine content to meet the regulatory requirements for the used oils. Method B covers the range from 200 to 4000 ppm of total chlorine, with a lower limit of quantitation estimated to be 870 to 1180 ppb.

TEST SUMMARY

The oil sample is dispersed in a hydrocarbon solvent and reacted with a mixture of metallic sodium catalyzed with napthalene and diglyme at ambient temperature. This converts organic halogens to their respective sodium halides which are then extracted into an aqueous buffer, which isthen titrated with mercuric nitrate using diphenyl carbazone as the indicator. The end point of the titration is the formation of the blue-violet mercury diphenylcarbazone complex.

Method A uses preset reagent quantities so that the final result clearly indicates pass or fail of the 1000 ppm limit.

TEST PRECISION

Repeatability:	$46.1\ X^{0.25}$
Reproducibility:	$84.44\ X^{0.25}$

Where X is the method result in mg/kg.

No bias statement for this test method can be made.

CHLORINE ORGANIC CHLORIDE IN CRUDE OIL: D 4929

EXPLANATION

Organic chloride species are potentially damaging to refinery processes. Usually they are not known to be naturally present in crude oils and usually result from cleaning operations at producing sites, pipelines, or tanks. Hydrochloric acid can be produced in hydrotreating or reforming reactors and the acid accumulates in condensing regions of the refinery. Unexpected amounts of organic chlorides cannot be effectively neutralized and damage can result.

This test method determines organic chloride above 1 ppm levels in crude oils using distillation followed by either sodium biphenyl reduction and potentiometric titration, or microcoulometry

TEST SUMMARY

Crude oil sample is distilled to obtain the naptha cut at 204°C (400°F). The naptha cut is washed successively with caustic and water to remove hydrogen sulfide and inorganic chlorides. This washed naptha fraction can be analyzed by two different procedures.

Method A—The washed naptha fraction is extracted with a solution of sodium biphenyl in toluene. The excess reagent is decomposed, the mixture acidified, and the phases separated. The aqueous phase is evaporated to a smaller volume and is potentiometrically titrated with standard silver nitrate solution.

Method B—The washed naptha fraction is injected into an oxidative furnace, and the resultant chlorides and oxychlorides are coulometrically titrated with silver nitrate.

Interferences—In Method A, HI and HBr will also titrate and give a positive response. In Method B, HI and HBr will also titrate and give a positive response. HOBr and HOI do not react in the titration cell, approximately 50 % microequivalent response is detected. In both methods total sulfur concentration of up to 10 000 times the chlorine level can be tolerated.

TEST PRECISION

	Repeatability	Reproducibility
Test Method A	$0.32(X+0.33)^{0.644}$	$0.7(X+0.33)^{0.644}$
Test Method B	$1.01(X-0.17)^{0.467}$	$1.32(X-0.17)^{0.467}$

All estimates are for chlorine level above 1 ppm. The method gives lower organic chloride values than present in the crude oil. This is because not all of the volatile components will distill from a complex crude oil under the distillation conditions of this method.

CLOUD POINT: D 5771/D 5772/D 5773

EXPLANATION

These three test methods are alternative procedures with automatic apparatus to Test Method D 2500 (see next page). When specifications quote D 2500, do not substitute these test methods without obtaining comparative data and customer agreement. All three methods are more precise than Test Method D 2500. All three test methods cover the range from −40+49°C with a temperature resolution of 0.1°C.

D 5771—ISL Instrument

After insertion of the specimen into the apparatus, and the initiation of the program, the specimen is cooled according to the cooling profile listed. The specimen is continuously monitored by an optical system for the formation of a crystalline structure. When the crystallization of the wax in the specimen is detected by the optical system, the temperature is recorded to within 0.1°C resolution. The specimen is then heated to facilitate the start of the next test.

D 5772—Herzog Instrument

After insertion of the specimen into the apparatus and initiation of the program, the specimen is heated and then cooled at a specified rate. The specimen is continuously monitored by an optical light barrier for the crystal structure formation. The temperature when the crystallization of the wax in the specimen is detected by the optical barrier is recorded with a resolution of 0.1°C. The specimen is then heated to the original starting temperature.

D 5773—Phase Technology Instrument

A specimen is cooled by a Peltier device at a constant rate of

1.5 ± 0.1°C/min while continuously being illuminated by a light source. The specimen is continuously monitored by an array of optical detectors for the first appearance of a cloud of wax crystals. The detectors are sufficient in number to ensure that any solid phase hydrocarbon crystals that may form are detected. The temperature at which the appearance of a cloud of wax crystals is first detected in the specimen is recorded to 0.1°C resolution. When the recorded temperature is rounded to the next lower integer temperature, it is designated as the D 2500/IP 219 equivalent cloud point per Test Method D 5773.

TEST PRECISION

Test Method	Repeatability, °C	Reproducibility, °C	Bias versus D 2500, °C
D 5771	2	3.1	−0.56
D 5772	0.8	3.7	−0.67
D 5773	0.5	2.6	−0.03

CLOUD POINT OF PETROLEUM PRODUCTS: D 2500

(Equivalent Test Methods: IP 219, ISO 3015, DIN 51597, JIS K 2269, and AFNOR T60-105)

EXPLANATION

The cloud point of a petroleum oil is an index of the lowest temperature of its utility for certain applications. Cloud point is the temperature at which a cloud of wax crystals first appears in a liquid when it is cooled under conditions prescribed in this test method. This test method covers only petroleum oils which are transparent in layers 38 mm (1 1/2 in.) in thickness, and with a cloud point below 49°C (120°F). The cloud point is usually higher than the pour point (Test Method D 97).

TEST SUMMARY

The sample is first heated to a temperature above the expected cloud point and then cooled at a specified rate and examined periodically. The temperature at which haziness is first observed at the bottom of the test jar is recorded as the cloud point.

TEST PRECISION

	Repeatability	Reproducibility
Distillate Oils	2°C(4°F)	4°C(8 °F)
Biodiesel blends in diesel	2°C(10°F)	3°C(10°F)

COEFFICIENT OF FRICTION OF LUBRICANTS: D 5183

EXPLANATION

This test method can be used to determine the coefficient of friction of lubricating fluids under the prescribed test conditions using a Four-Ball Wear Test Machine.

TEST SUMMARY

Three steel balls are clamped together and covered with wear-in lubricant. A fourth ball is pressed with a force of 392 N into the cavity formed by the three clamped balls for three point contact. The temperature of the lubricant is regulated at 75°C, and the fourth ball is rotated at 600 rpm for 60 min. At the end, the fluid is discarded and the balls cleaned. The wear scar diameter on each of the lower three balls is examined. If the wear scar is 0.67 mm, then successively more oil is added, and the ball is rotated for 10 min each time. The coefficient of friction is measured at the end of each 10 min interval.

TEST PRECISION

Repeatability:	0.20 X
Reproducibility:	0.49 X

Where X is the average of successive results.

Bias of this test method has not been determined.

FRICTION AND WEAR PROPERTIES OF LUBRICATING GREASE
D 5707

EXPLANATION

This test method is used for determining wear properties and coefficient of friction of lubricating greases at selected temperatures and loads specified for use in applications where high-speed vibrational or start-stop motions are present for extended periods of time under initial high Hertzian point contact pressures. This test method has found application in qualifying lubricating greases used in constant velocity joints of front-wheel-drive automobiles and lubricating greases used in roller bearings.

TEST SUMMARY

This test method is performed on an SRV test machine using a test ball oscillated under constant load against a test disk. Following conditions are used: 200 N test load, 50 Hz frequency, 1.00 mm stroke amplitude, 2-h duration, and temperature of ambient to 280°C. Other test loads (10 to 1400 N), frequencies (5 to 500 Hz, and stroke amplitudes (0.1 to 3.30 mm) can be used, if specified. The wear scar on the test ball and coefficient of friction are measured. If a profilometer is available, a trace of the wear scar on the disk can also be used to obtain additional wear information.

TEST PRECISION

Measurement	Repeatability	Reproducibility
Average Ball Wear Scar Diameter	0.07 mm	0.29 mm (at 50°C) 0.24 mm (at 80°C)
Minimum Coefficient of Friction at 50°C	0.012	0.031
at 80°C	0.008	0.032

This test method has no bias.

COEFFICIENT OF KINETIC FRICTION FOR WAX COATINGS:
D 2534

EXPLANATION

The coefficient of friction is related to the slip properties of many commercial articles which have been coated with petroleum wax (e.g., butter cartons and wax impregnated or hot melt coated corrugated board). This test method covers the determination of the coefficient of kinetic friction for a petroleum wax coating or wax-based hot melt coating when sliding over itself.

TEST SUMMARY

A coated surface under load is pulled at a uniform rate over a second coated surface. The force required to move the load is measured, and the coefficient of kinetic friction is calculated.

TEST PRECISION

Repeatability:	$0.06\mu_k$
Reproducibility:	$0.17\mu_k$

In the range from 0 to 1.

This test method has no bias because the value of coefficient of friction can be defined only in terms of this test method.

COKING VALUE OF TAR AND PITCH
D 4715

EXPLANATION

This test method is used for indicating the relative coke-forming properties and for evaluating and characterizing tars and pitches. This is one of the elements in establishing uniformity of shipments of sources of supply. This test method is applicable to tars and pitches having an ash content not over 0.5 % as determined by Test Method D 2415. Coking values by this method are higher than those obtained by Test Method D 2416.

TEST SUMMARY

A test portion of tar or pitch is heated for a specified time at $1022 \pm 18°F(550 \pm 10°C)$ in an electric furnace. The percentage of residue is reported as the coking value.

TEST PRECISION

Repeatability:	1.2 % absolute
Reproducibility:	2.3 % absolute

The coking values by this test method are about two percentage points higher than those obtained by Test Method D 2416.

COLD CRANKING SIMULATOR APPARENT VISCOSITY USING COLD CRANKING SIMULATOR:
D 2602
(Equivalent Test Methods: IP 350 and DIN 51377)

EXPLANATION

The apparent viscosity as determined in this test method has been established as predicting engine-cranking viscosities at −18°C. This test method covers the laboratory determination of apparent viscosity of engine oils at 0°F(−17.8°C) and at high shear rates. The results are related to engine-cranking characteristics of engine oils. Since this test method was developed solely for use in relation to engine cranking characteristics of engine oils, the apparent viscosity values obtained should not be used to predict other types of performance. For example, this test method is not suitable to predict flow rates to rocker arm bearings, or other engine locations at low temperatures.

TEST SUMMARY

A universal motor drives a rotor which is closely fitted inside a stator. A small sample of engine oil fills the space between rotor and stator which are maintained at 0°F(−17°C). The speed of the rotor is a function of the viscosity of the oil. From a calibration curve and the measured speed of the rotor with the oil under test, the viscosity of the test oil is determined.

TEST PRECISION

Repeatability:	6 % of the mean
Reproducibility:	12 % of the mean

No bias statement can be made.

COLD FILTER PLUGGING POINT (CFPP) OF DIESEL AND HEATING FUELS:
D 6371
(Equivalent Test Methods: IP 309 and EN 116)

EXPLANATION

The CFPP of a fuel is suitable for estimating the lowest temperature at which a fuel will give trouble-free flow in certain fuel systems. For diesel fuels used in European light duty trucks, the results are usually close to the temperature of failure in service except in some specific circumstances. Domestic heating installations are usually less critical and often operate satisfactorily at temperatures somewhat lower than those indicated by the test results. The difference in results obtained from the sample as received and after heat treatment at 45°C for 30 min can be used to investigate complaints of unsatisfactory performance under low temperature conditions.

This test method is applicable to distillate fuels including those containing a flow-improving or other additive intended for use in diesel engines and domestic heating installations.

TEST SUMMARY

Either manual or automated apparatus may be used. A sample is cooled under specified conditions and at intervals of 1°C, is drawn into a pipette under a controlled vacuum through a standardized wire mesh filter. As the sample continues to cool, the procedure is repeated for each 1°C below the first test temperature. The testing is continued until the amount of wax crystals that have separated out of the solution is sufficient to stop or slow down the flow so that the time taken to fill the pipet exceeds 60 s or the fuel fails to return completely to the test jar before the fuel has cooled by a further 1°C. The indicated temperature at which the last filtration was commenced is recorded as the CFPP.

TEST PRECISION

Repeatability:	1.76°C
Reproducibility:	$0.102(25-X)$°C

Where X is the average of the two results.

There is no bias. There is no relative bias between manual and automated apparatus.

COLOR ASTM COLOR:
D 1500
(Equivalent Test Methods: IP 196, ISO 2049, DIN 51578, JIS K 2580, and AFNOR T60-104)

GENERAL

Determination of the color of petroleum products is used mainly for manufacturing control purposes and is an important quality characteristic since color is readily observed by the user of the product. In some cases the color may serve as an indication of the degree of refinement of the material. When the color range of a particular product is known, a variation outside the established range may indicate possible contamination with another product. However, color is not always a reliable guide to product quality and should not be used indiscriminately in product specifications.

Several color scales are used for determination:

ASTM Color	D 1500
Gardner Color	D 1544
Platinum-Cobalt Color	D 1209
Saybolt Color	D 156

Comparison of color values obtained by different methods is shown in Fig. 3.

EXPLANATION

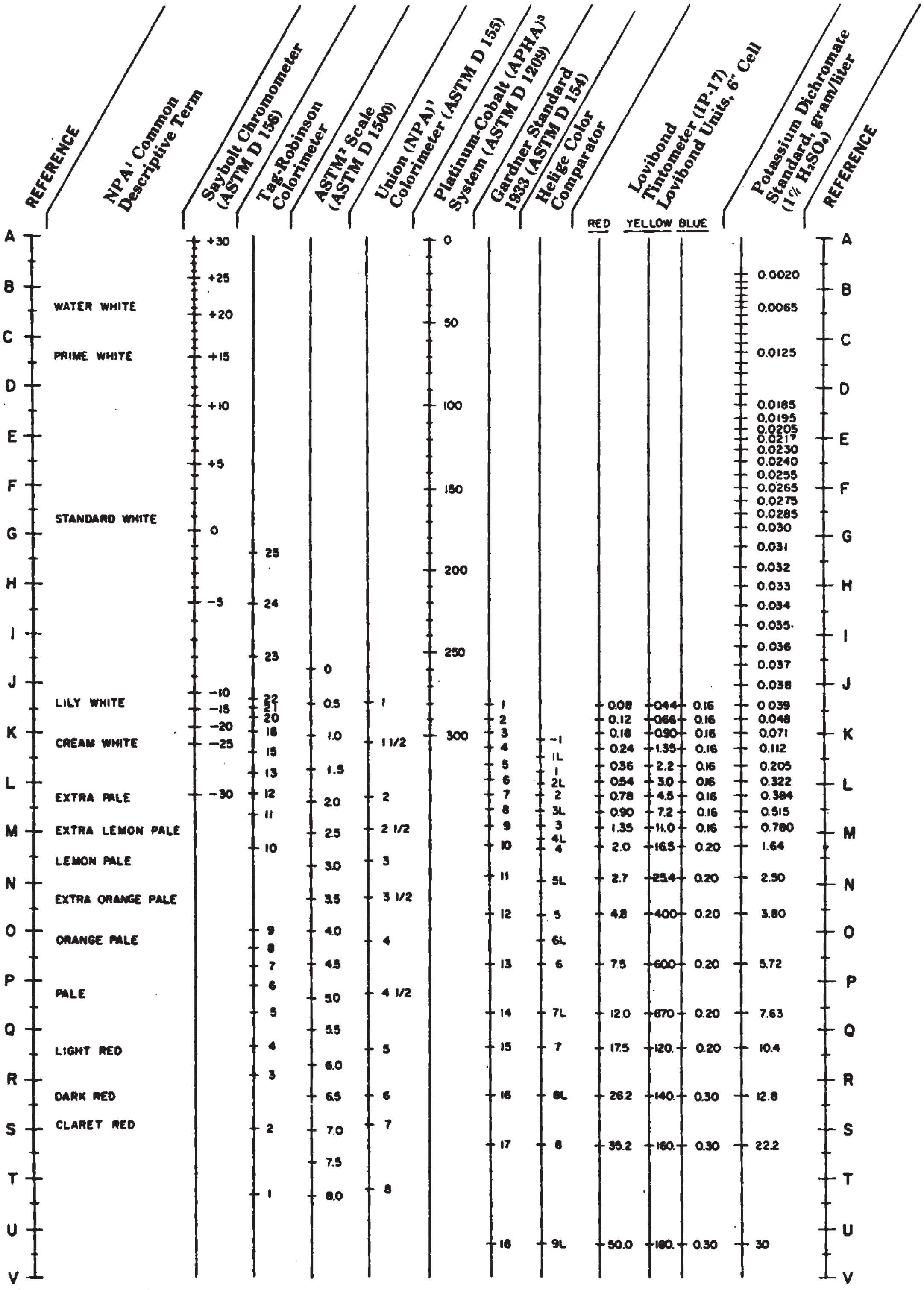
This test method covers the visual determination of the color of a wide variety of petroleum products such as lubricating oils, heating oils, diesel fuel oils, and petroleum waxes. Test Method D 156 is applicable to refined products that have an ASTM color lighter than 0.5.

TEST SUMMARY

Using a standard light source, a liquid sample is placed in the test container and compared with colored glass disks ranging in value from 0.5 to 8.0. When an exact match is not found and the sample color falls between two standard colors, the higher of the two colors is reported.

TEST PRECISION

Repeatability:	0.5
Reproducibility:	1.0



NOTES
(1) NATIONAL PETROLEUM ASSOCIATION
(2) AMERICAN SOCIETY FOR TESTING AND MATERIALS
(3) AMERICAN PUBLIC HEALTH ASSOCIATION

FIG. 3—*Color scale comparisons for approximate conversions.*

COLOR OF DYED AVIATION GASOLINE: D 2392

EXPLANATION

Aviation gasolines are dyed different colors for easy identification of grade, thus minimizing possibilities for fueling aircraft with fuel of the wrong grade. This test method is a pass-fail test for the acceptability of color of dyed aviation gasolines.

TEST SUMMARY

The hue and color intensity of a sample of the gasoline to be tested are compared with those of glass permanent color standards.

TEST PRECISION

Since this is a pass-fail test, no statement of precision or bias can be made.

COLOR GARDNER COLOR: D 1544

EXPLANATION

This test method covers the measurement of the color of transparent liquids by means of comparison with arbitrarily numbered glass standards. It applies to drying oils, varnishes, fatty acids, polymerized fatty acids, and resin solutions.

TEST SUMMARY

A glass tube is filled with sample and under illumination, is compared with glass standards, comparing which standard most closely matches the sample in brightness and saturation.

TEST PRECISION

Repeatability:	0.66 units
Reproducibility:	2.0 units

COLOR PLATINUM-COBALT COLOR: D 1209

EXPLANATION

This test method describes a procedure for the visual measurement of the color of essentially light colored liquids. It is applicable only to materials in which the color-producing bodies present have light absorption characteristics nearly identical with those of the platinum-cobalt color standards used. Sometimes this test is referred to as "APHA [American Public Health Association] Color." The petroleum industry uses the Saybolt colorimeter Test Method D 156 for measuring and defining the color of hydrocarbon solvents; however, this system of color measurement is not commonly employed outside of the petroleum industry. It has been reported by various sources that a Saybolt color of +25 is equivalent to 25 in the platinum-cobalt system or to colors produced by masses of potassium dichromate ranging between 4.8 and 5.6 mg dissolved in 1 L of distilled water. Because of the differences in the spectral characteristics of the several color systems being compared and the subjective manner in which the measurements are made, exact equivalencies are difficult to obtain.

TEST SUMMARY

One hundred mL of sample is placed in a Nessler tube. The sample is passed through a filter if it has any visible turbidity. The tube is placed in a comparator and compared with the standards.

TEST PRECISION

Repeatability:	2
Reproducibility:	7

COLOR SAYBOLT COLOR:
D 156
(Equivalent Test Methods: DIN 51411, JIS K 2580, and AFNOR M07-003)

EXPLANATION

Saybolt Color—An empirical definition of the color of a clear petroleum liquid based on a scale of −16 (darkest) to +30 (lightest). The number is derived by finding the height of a column of the sample that, when viewed through the length of the column, visually matches the appropriate one of three glass standards.

This test method covers the determination of the color of refined oils such as undyed motor and aviation gasoline, jet propulsion fuels, naphthas and kerosene, and, in addition, petroleum waxes and pharmaceutical white oils. For determining the color of petroleum products darker than Saybolt Color −16, see Test Method D 1500.

TEST SUMMARY

The height of a column of sample is decreased by levels corresponding to color numbers until the color of the sample is unmistakably lighter than that of the standard. The color number above this level is reported, regardless of whether the sample was darker, questionable, or a match at the higher level.

TEST PRECISION

Repeatability:	1 color unit
Reproducibility:	2 color unit

COLOR BY AUTOMATIC TRISTIMULUS METHOD:
D 6045

EXPLANATION

Determination of the color of petroleum products is used mainly for manufacturing control purposes and is an important quality characteristic because the color is readily observed by the user of the product. In some cases the color may serve as an indicator of the degree of refinement of the material. A variation outside the color range of a product may indicate possible contamination with another product. However, color is not always a reliable guide to product quality and should not be used indiscriminately in product specifications.

This test method covers the automatic determination of color of a wide variety of petroleum products such as undyed motor and aviation gasoline, aviation turbine fuels, napthas, kerosene, pharmaceutical white oils, diesel fuel oils, and lubricating oils. With appropriate sample handling, this test method would apply to petroleum waxes, but they were not included in the round robin, and the precision of this method with regard to waxes is unknown. This test method does not apply to solid samples, petroleum products containing dyes or having extreme fluorescence, or to cloudy samples. In the last case, the samples shall be filtered so they are clear before measuring.

This test method has one-to-one correlation for the entire range of Test Method D 1500, ASTM color and for the range from 0 to +30 for Test Method D 156, Saybolt color.

TEST SUMMARY

The sample is poured into a glass sample container, and the container is placed into the light path of the automatic instrument. A transmittance measurement is made in order to determine the CIE tristimulus values of the sample. These are then converted instrumentally by the appropriate algorithm to Saybolt or ASTM color values.

TEST PRECISION

Repeatability:	0.14 Saybolt color units 0.10 ASTM color units
Reproducibility:	1.24 Saybolt color units 0.48 ASTM color units

Bias—There is no bias between this test method and Test Method D 1500, ASTM color method. There is no bias between this test method and Test Method D 156 Saybolt color method in the range from 0 to +30; however, there is bias for the range from 0 to −16.

CONE PENETRATION OF LUBRICATING GREASES:
D 217
(Equivalent Test Methods: IP 50, ISO 2137, and AFNOR T60-132)

EXPLANATION

Cone penetration test provides one measure of the consistency of a grease. These tests cover the full range of NLGI numbers from 000 to 6. They also evaluate the consistency of stiff greases with penetration numbers less than 85. Although no correlation has been developed between cone penetration results and field service, the former results are widely used for specification purposes in users' and suppliers' manufacturing specifications.

Test Method D 937 is aimed at petrolatums, and Test Method D 1403 is used for limited sample quantities.

TEST SUMMARY

This test method consists of four procedures. For unworked penetration, the cone assembly of the penetrometer is allowed to drop freely into the grease sample at 25°C in a worker cup for 5 s. For worked penetration, the sample at 25°C in a worker cup is subjected to 60 double strokes by the grease worker. The penetration is determined immediately by releasing the cone assembly from the penetrometer and allowing the cone to drop freely into the grease for 5 s. For prolonged work penetration, the procedure is the same as for worked penetration, except that additionally before cone penetration, the sample is subjected to a predetermined number of double strokes in the grease worker. For block penetration, a cube of grease is used and the test is followed as in that for unworked penetration.

In all cases three determinations are made and the average value is reported.

TEST PRECISION

Penetration	Range	Repeatability	Reproducibility
Unworked	85–475	8	19
Worked	130–475	7	20
Prolonged Worked	130–475	15[a]	27[a]
Block	under 85	7	11

[a] Value is determined at 100 000 double strokes within 15 to 30°C ambient temperature range.

CONE PENETRATION OF LUBRICATING GREASE:
D 1403
(Equivalent Tests: IP 310 and AFNOR T60-140)

EXPLANATION

See discussion under Test Method D 217. The current method is intended for use only when the size of the sample is limited. Precision is better in the full scale method.

These test methods include procedures for the measurement of unworked and worked penetrations. Unworked penetrations do not generally represent the consistency of greases in use as effectively as do worked penetrations. The latter are usually preferred for inspecting lubricating greases.

TEST SUMMARY

There are two test procedures, one for one-fourth scale cone and the other for the half scale cone. The penetration is determined at 25°C(77°F) by releasing the cone assembly from the penetrometer and allowing the cone to drop freely into the grease for 5 s.

TEST PRECISION

Penetrations	Repeatability	Reproducibility
Unworked: $\frac{1}{4}$ scale	3	10
$\frac{1}{2}$ scale	5	13
Worked: $\frac{1}{4}$ scale	3	7
$\frac{1}{2}$ scale	3	10

The bias of this test method has not been determined.

CONE PENETRATION OF PETROLATUM:
D 937
(Equivalent Tests: IP 179, DIN 51580, and AFNOR T60-119)

EXPLANATION

Petrolatum is a purified mixture of semi-solid hydrocarbons obtained from petroleum and is often described as an unctuous mass. Cone penetration is a means of measuring the firmness of consistency of petrolatum. Such measurements are used in selecting or specifying a petrolatum, or both, of a particular consistency or firmness. Cone penetration values may or may not correlate with end-use functional properties.

TEST SUMMARY

The sample is melted, heated to 82°C, and then cooled under controlled conditions to 25°C. The penetration is measured with the cone and the sample at this temperature using a penetrometer by means of which a standard cone is applied to the sample for 5 s under a load of 150 g.

TEST PRECISION

Repeatability:	2+5 % penetration value
Reproducibility:	9+12 % penetration value

This test method has no bias.

CONGEALING POINT OF PETROLEUM WAXES AND PETROLATUM:
D 938
(Equivalent Tests: IP 76, ISO 2207, and AFNOR T60-128)

EXPLANATION

Congealing point is a wax property of interest to many petroleum wax consumers. This procedure measures the temperature at which a sample being cooled develops a “set” or resistance to flow. At that temperature the wax may be at or close to the solid state, or it may be semisolid and quite unctuous, depending on the composition of the wax or petrolatum being tested. In the case of petrolatums, congealing property is associated with the formation of a gel structure as the sample cools.

TEST SUMMARY

A droplet of the melted wax sample is made to adhere to the bulb of a thermometer. Using a prewarmed flask as an air jacket, the droplet on the bulb is allowed to cool at a fixed rate until it congeals. The congealing point is observed as the temperature at which the droplet ceases to flow as the thermometer is turned.

TEST PRECISION

Samples	Repeatability	Reproducibility
Distillate Waxes	0.5°C(1.0°F)	1.5°C(2.5°F)
Residual Waxes Including Petrolatum	1.0°C(2.0°F)	2.5°C(4.5°F)

This test method has no bias.

CONRADSON CARBON RESIDUE OF PETROLEUM PRODUCTS:

D 189

(Equivalent Test Methods: IP 13, ISO 6615, DIN 51551, JIS K 2270, and AFNOR T60-116)

EXPLANATION

The carbon residue value of burner fuel serves as a rough approximation of the tendency of the fuel to form deposits in vaporizing pot-type and sleeve-type burners. Similarly, provided amyl nitrate is absent (or if it is present, provided the test is performed on the base fuel without additive) the carbon

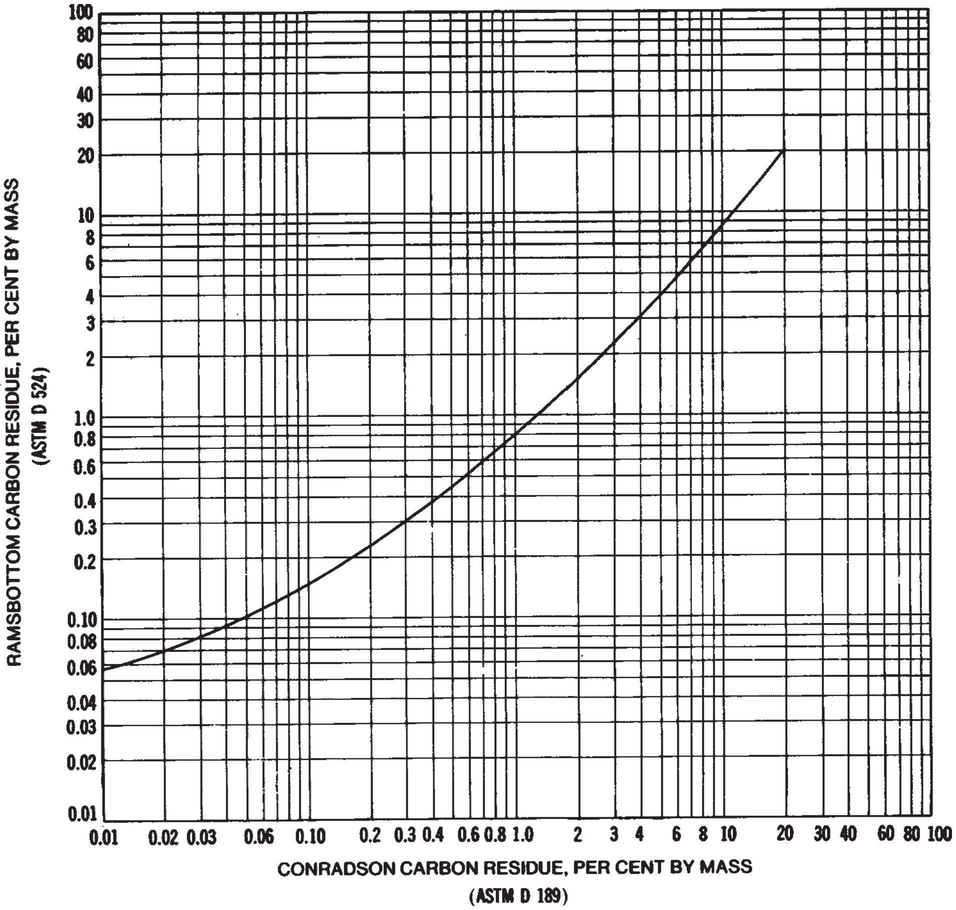


FIG. 4—*Precision.*

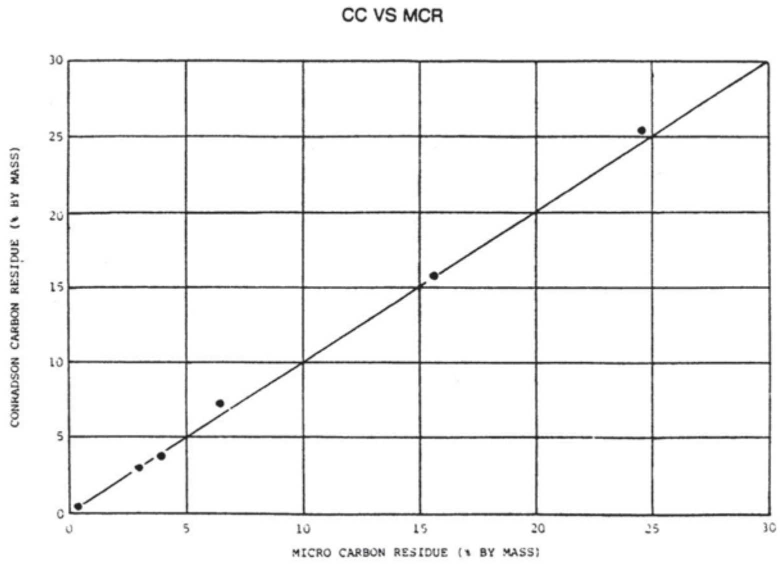


FIG. 5—*Correlation between D 189 and D 524 Test Methods.*

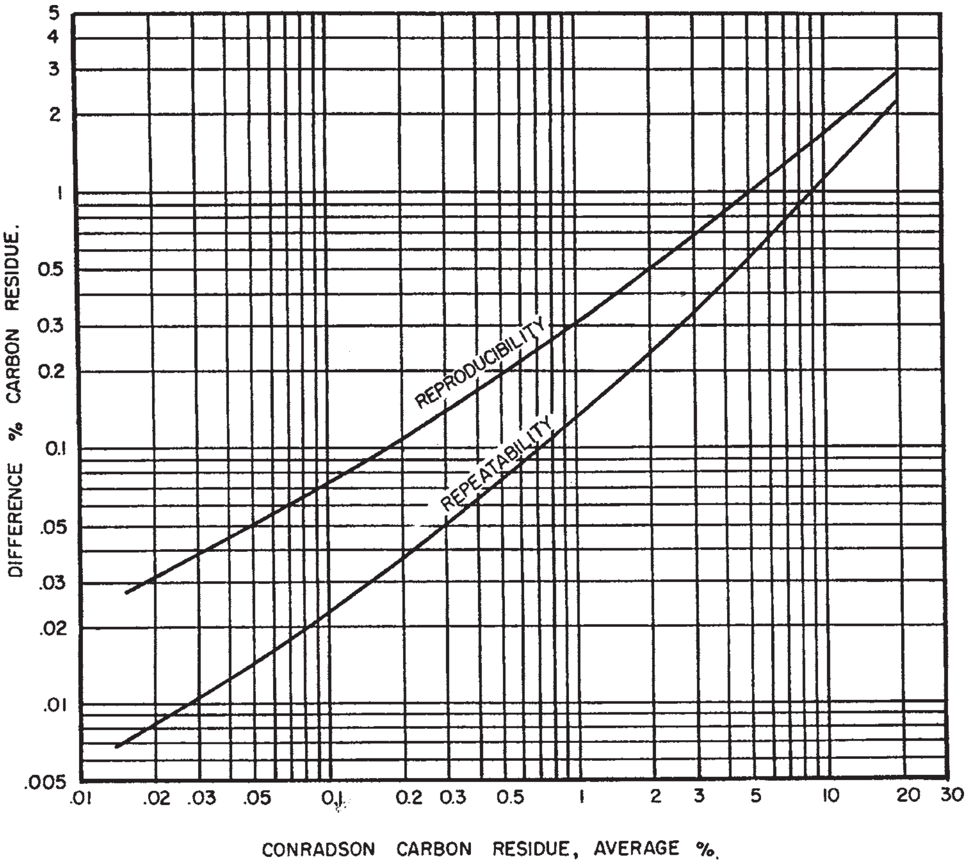


FIG. 6—*Correlation between D 189 and D 4530 Test Methods.*

residue of diesel fuel correlates approximately with combustion chamber deposits.

The carbon residue value of motor oil, while at one time regarded as indicative of the amount of carbonaceous deposits a motor oil would form in the combustion chamber of an engine, is now considered to be of doubtful significance due to the presence of additives in many oils. For example, an ash-forming detergent additive may increase the carbon residue value of an oil yet will generally reduce its tendency to form deposits.

This test method covers the determination of the amount of carbon residue left after evaporation and pyrolysis of an oil, and is intended to provide some indication of relative coke-forming propensities. This test method is generally applicable to relatively nonvolatile petroleum products, which partially decompose on distillation at atmospheric pressure. Petroleum products containing ash-forming constituents will have an erroneously high carbon residue, depending upon the amount of ash formed.

TEST SUMMARY

A weighed quantity of sample is placed in a crucible and subjected to destructive distillation. The residue undergoes cracking and coking reactions during a fixed period of severe heating. At the end of the specified heating period, the test crucible containing the carbonaceous residue is cooled in a desiccator and weighed. The residue remaining is calculated as a percentage of the original sample, and reported as Conradson carbon residue.

TEST PRECISION

Repeatability and reproducibility of Test Method D 189 should be within limits shown in Fig. 4.

CORRELATION WITH OTHER TESTS

No exact correlation between results of D 189 and D 524 exists because of the empirical nature of the two tests. However, an approximate correlation (Fig. 5) has been derived between these two methods. Also, Fig. 6 shows a direct correlation obtained between D 189 and D 4530 test methods.

CONRADSON CARBON RESIDUE COKING VALUE OF TAR AND PITCH:
D 2416

EXPLANATION

This test method is useful for indicating the relative coke-forming propensities and for evaluating and characterizing tars and pitches. This test method can also be used as one element in establishing the uniformity of shipments or supply sources. This test method is applicable to samples with an ash content not over 0.5 % as determined by Test Method D 2415.

Coking values obtained by this test method are practically the same as those obtained by Test Method D 189, Conradson carbon, but the results are more reproducible. The apparatus used is identical, except that an electric furnace is substituted for gas flame.

TEST SUMMARY

A sample of tar or pitch is vaporized and pyrolized for a specified time at a specified temperature in special standardized equipment that limits the available oxygen supply. The percentage of residue is reported as the coking value.

TEST PRECISION

Repeatability:	1.0
Reproducibility:	2.0

COOLING CHARACTERISTICS OF QUENCH OILS BY COOLING CURVE ANALYSIS:
D 6200

EXPLANATION

This test method provides a cooling time versus temperature pathway which is directly proportional to physical properties such as the hardness obtainable upon quenching of a metal. These results may be used as a guide in heat treating oil selection or comparison of quench severities of different heat treating of new or used oils. This test is designed to evaluate quenching oils in a nonagitated system. There is no correlation between these test results and the results obtained in agitated systems.

TEST METHOD SUMMARY

The nickel alloy probes assembly's cooling time versus temperature is determined after placing the assembly in a furnace and heating to 850°C, and then quenching in a heat treating oil. The temperature inside the probe assembly and the cooling times are recorded at selected time intervals to establish a cooling temperature versus time curve. The resulting cooling curve may be used to evaluate quench severity.

The presence of water in a heat treating oil has a major effect upon the results obtained with this test method. If the water content is above 0.01 %, the calibration fluid has to be dried at a minimum temperature of 102°C until water content is at or below 0.01 %. The water content may be determined using the Test Method D 1744.

TEST PRECISION

Parameter	Range of Oils	Repeatability	Reproducibility
Maximum Cooling Rate	47 to 98°C/s	2.1°C/s	8.6°C/s
Temperature of Maximum Cooling Rate	490 to 590°C	12.7°C	25.3°C
Cooling Rate at 300°C	5.8 to 33.4°C/s	8.7 % of Mean	25 % of Mean
Time to Cool to 600°C	8.6 to 13.4 s	0.4 s	1.4 s
Time to Cool to 400°C	11.2 to 22.5 s	0.5 s	2.1 s
Time to Cool to 200°C	27.2 to 59.7 s	1.3 s	10.1 s

This test method has no bias because the cooling characteristics can be defined only in terms of this test method.

COPPER IN JET FUELS BY GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY:
D 6732

EXPLANATION

At high temperatures aviation turbine fuels can oxidize and produce insoluble deposits that are detrimental to aircraft propulsion systems. Very low copper levels in excess of 50 μg/kg can significantly accelerate this thermal instability. Since naval shipboard aviation fuel delivery systems contain copper-nickel piping this can increase the copper levels in the fuel. Thus, this method can be used to check the copper levels in aviation turbine fuel samples taken on shipboard, in refineries, and at fuel storage depots. This test method covers the copper range of 5–100 μg/kg. Copper concentration above this level may also be determined by diluting the samples with kerosene to bring the copper level in the above range.

TEST METHOD SUMMARY

Typically 10 μL of the sample is pipetted onto a platform in the graphite furnace. The furnace is heated to low temperature to dry the sample completely without spattering. It is next heated to moderate temperature to eliminate excess sample matrix, and finally heated very rapidly to a temperature high enough to volatilize copper. In this step the amount of light absorbed by the copper atoms is measured by the spectrometer over a specified period of time. The integrated absorbance produced by copper in the sample is compared to a calibration curve constructed from the measured value for organo-metallic standards.

The char step in the heating program is used to eliminate as much of the matrix as possible before the atomization step to reduce undissociated molecular particles from the sample matrix. Most spectrometers are also equipped with background correction capabilities to further control possibilities of erroneous results due to molecular absorption.

TEST PRECISION

Repeatability:	$(X+1)^{0.5}$
Reproducibility:	$4.5(X+1)^{0.5}$

Where X is the average of two results in μg/kg.

No statement regarding bias of this test method can be made since there is no accepted reference material for this analysis.

COPPER CORROSION: D 130 (Equivalent Test Methods: IP 154, ISO 2160, DIN 51759, JIS K 2513, and AFNOR M07-015)

GENERAL

These test methods determine the corrosiveness to copper of various gasolines, fuels, lubricating oils or other hydrocarbons. Most sulfur compounds in petroleum are removed during refining. However, some residual sulfur compounds can have a corroding action on various metals. This effect is dependent on the types of sulfur compounds present. The copper strip corrosion test measures the relative degree of corrosivity of a petroleum product.

The three available methods for this analysis are described here.

TEST SUMMARY

A polished copper strip is immersed in a given quantity of sample and heated at a temperature for a time period characteristic of the material being tested. At the end of this period the copper strip is removed, washed and compared with the ASTM copper strip corrosion standards.

TEST PRECISION

This is a pass/fail test, and no precision data are available.

COPPER CORROSION: D 849

TEST SUMMARY

A polished copper strip is immersed in 200 mL of specimen in a flask with a condenser and placed in boiling water for 30 min. At the end of this period, the copper strip is removed and compared with the ASTM copper strip corrosion standards.

TEST PRECISION

This is a pass-fail test, and no precision data are available.

COPPER STRIP CORROSION BY LIQUEFIED PETROLEUM GASES:
D 1838

EXPLANATION

Copper corrosion limits provide assurance that difficulties will not be experienced in deterioration of the copper and copper-alloy fittings and connections that are commonly used in many types of utilization, storage, and transportation equipment. For an equivalent copper strip test applicable to less volatile petroleum products Test Method D 130 may be used.

TEST SUMMARY

A polished copper strip is immersed in approximately 100 mL of the sample and exposed at a temperature of 37.8°C for one hour in a cylinder of suitable working pressure. At the end of this period, the copper strip is removed and rated as one of the four classifications of the ASTM Copper Corrosion Standards.

TEST PRECISION

No generally accepted method for determining precision or bias of ordinal classification data is currently available.

COPPER CORROSION FROM LUBRICATING GREASE:
D 4048

EXPLANATION

This test method measures the tendency of lubricating grease to corrode copper under specific static conditions. It can help in predicting possible chemical attack on lubricating parts such as bearings containing copper or copper alloys. Such corrosion can cause premature bearing failures. However, no correlation with actual field service has been established. The test does not measure either the ability of the lubricant to inhibit copper corrosion caused by factors other than the lubricant itself nor does it measure the stability of the grease in the presence of copper.

TEST SUMMARY

A prepared copper strip is totally immersed in a sample of grease and heated in an oven or liquid bath at a specific temperature for a definite period of time. Commonly used conditions are 100°C for 24 h. At the end of this heating period, the strip is removed, washed, and compared with the ASTM copper strip corrosion standards.

TEST PRECISION

Because of the nature of this test, the precision of this test method was not obtained in accordance with standard ASTM protocol. The test has no bias.

CORROSIVENESS TO COPPER FROM PETROLEUM PRODUCTS:
D 7095

EXPLANATION

Crude petroleum contains sulfur compounds, most of which are removed during refining. However, of the remaining sulfur compounds can have a corroding action on various metals, including copper, and this corrosivity is not necessarily related to the sulfur content. The effect can vary according to the chemical types of sulfur compounds present. This copper strip corrosion test is designed to assess the relative degree of corrosivity of a petroleum product towards copper and copper-containing alloys using a shorter test duration than that specified in Test Method D 130. Some sulfur species may become corrosive to copper only at higher temperatures. Thus, higher test temperatures, particularly 100°C, may be used to test some products by the pressure vessel procedure.

This test method is applicable to aviation gasoline, aviation turbine fuel, automotive gasoline, natural gasoline, or other hydrocarbons having a vapor pressure not greater than 124 kPa, cleaners such as Stoddard solvent, kerosene, diesel fuel, distillate fuel oil, lubricating oil, and other petroleum products.

This test method is similar to Test Method D 130, but involves three major differences: (1) A single-use copper foil strip is used in place of Test Method D 1340 multi-use copper strip. (2) A different polishing technique is used in preparing the copper foil strip. (3) This test method involves a shorter analysis time of 45 minutes for all product types versus two to three hours required in Test Method D 130.

TEST METHOD SUMMARY

A polished copper strip is immersed in a specific volume of the sample being tested and heated under conditions of temperature and time that are specific to the class of material being tested. At the end of the heating period, the copper foil strip is removed, washed, and the color and tarnish level are assessed against the ASTM copper strip corrosion standard.

TEST PRECISION

In the case of pass/fail data, no generally accepted method for determining precision or bias is currently available.

CORROSION OF SOLID FILM LUBRICANTS: **D 2649**

EXPLANATION

This test method evaluates the corrosion characteristics of dry solid film lubricants under conditions of high humidity. Surfaces are examined for signs of corrosion, which is an indication of potential field failure.

TEST SUMMARY

An aluminum panel with a solid film deposited on one surface is contacted under load with the surface of an unlubricated panel. The specimen is subjected to 95±3 % relative humidity at 49°C(120°F) for a period of 500 h. At the end, the surface of the unlubricated panel is examined for the evidence of corrosion.

TEST PRECISION

This is a pass-fail test and normal precision or bias statements are not applicable.

CORROSIVENESS OF DIESEL OILS AT 135°C: **D 6594**

EXPLANATION

This test method simulates the corrosion process of non-ferrous metals (particularly alloys of lead and copper) in diesel lubricants. The corrosion process under investigation is believed to be induced primarily by inappropriate lubricant chemistry rather than lubricant degradation or contamination. This test method has been found to correlate with an extensive fleet database containing corrosion-induced cam and bearing failures.

TEST SUMMARY

Four metal specimens of copper, tin, lead, and phosphors bronze are immersed in a measured amount of engine oil. The oil at an elevated temperature is blown with air for a period of time. When the test is completed, the copper specimen and the stressed oil are examined to detect corrosion and corrosion products, respectively. A reference oil is tested with each group of tests to verify the test acceptability. The ASTM Test Monitoring Center (TMC) provides reference oils and an assessment of the test results obtained on these oils by the laboratory.

TEST PRECISION

Copper	Repeatability	$S_{I.P.}=0.318$	I.P. = 890
	Reproducibility	$S_R=0.349$	R = 0.977
Lead	Repeatability	$S_{I.P.}=15.54$	I.P. = 43.51
	Reproducibility	$S_R=17.16$	R = 48.05
Tin	Repeatability	Not Determined	
	Reproducibility	Not Determined	

Where I.P. is repeatability, and $S_{I.P.}$ is standard deviation.

Bias of this method is unknown at this time, but will be determined as more data are collected.

CORROSION PREVENTIVE PROPERTIES CORROSIVENESS AND OXIDATION STABILITY OF OILS:

D 4636

EXPLANATION

This test method is a combination of Federal Test Methods 5307.2 and 5308.7. It is used to test hydraulic oils, aircraft turbine engine lubricants, and other highly refined oils to determine their resistance to oxidation and corrosion degradation and their tendency to corrode various metals. Petroleum and synthetic fluids may be tested using moist or dry air with or without metal test specimens.

Interpretation of results should be done by comparison with data from oils of known field performance. This test is most suitable for oils containing oxidation and corrosion inhibitors. Without such ingredient(s) the severe test conditions will yield rather drastic changes to the oil.

TEST SUMMARY

This test method consists of one standard and two alternative procedures. A particular specification needs to establish which of these tests should be used. A large glass tube containing an oil sample and metal specimens is placed in a constant temperature bath (usually from 100 to 360°C) and heated for the specific number of hours while air is passed through the oil to provide agitation and a source of oxygen. Corrosiveness of the oil is determined by the loss in metal mass, and microscopic examination of the sample metal surface(s). Oil samples are withdrawn from the test oil and checked for changes in viscosity and acid number as a result of the oxidation reactions. At the end of the test the amount of the sludge present in the oil remaining in the same tube is determined by centrifugation. Also, the quantity of oil lost during the test is determined gravimetrically.

Metals used in the basic test and alternative test Procedure 1 are titanium, magnesium, steel (two types), bronze, silver, and aluminum. Metals used in alternative test Procedure 2 are copper, steel, aluminum, magnesium, and cadmium.

Other metals may be specified.

TEST PRECISION

The precision and bias of this test method is being balloted in the DO2 Committee.

CORROSIVENESS OF LUBRICATING FLUID TO BIMETALLIC COUPLE:
D 6547

EXPLANATION

Corrosiveness of a fluid to a bimetallic couple is one of the properties used to evaluate hydraulic or lubricating fluids. It is an indicator of the compatibility of a fluid with a brass on steel galvanic couple at ambient temperature and 50 % relative humidity. This test method replicates Fed-Std No. 791, method 5322.2. It utilizes the same apparatus, test conditions, and evaluation criteria, but it describes the test procedures more explicitly.

TEST SUMMARY

A brass clip is fitted to the fluid coated surface of a steel disk; the assembly is stored at approximately 50 % relative humidity for ten days and visually inspected for the evidence of galvanic corrosion.

TEST PRECISION

Since this is only a pass-fail test, it is not practical to specify the precision of this method.

The procedure in this test method has no bias because the value of corrosion-preventing characteristics is defined only in terms of this test method.

CORROSION PREVENTIVE PROPERTIES OF LUBRICATING GREASES:
D 1743

EXPLANATION

This test method differentiates the relative corrosion-preventing properties of lubricating greases using grease-lubricated tapered roller bearings stored under wet conditions. This test method is based on CRC Technique L 41 that shows correlation between laboratory results and service for grease lubricated aircraft wheel bearings.

TEST SUMMARY

New, cleaned, and lubricated bearings are run under a light thrust load for 60 s to distribute the lubricant in a pattern that might be found in service. The bearings are exposed to water, then stored for 48 h at 100 % relative humidity. After cleaning, the bearing cups are examined for evidence of corrosion.

TEST PRECISION

Due to the nature of the results, the precision of this test method has not been obtained by standard ASTM protocol.

Bias statement also does not apply to this test.

CORROSION-PREVENTING PROPERTIES OF LUBRICATING GREASES:
D 5969

EXPLANATION

This test method differentiates the relative corrosion-preventive capabilities of lubricating greases under the test conditions using grease lubricated tapered roller bearings exposed to various concentrations of dilute synthetic sea water stored under wet conditions. It is based on Test Method D 1743, which is practiced using a similar procedure and distilled water. The reported result is a pass or fail rating as determined by at least two of three bearings.

TEST SUMMARY

New, cleaned, and lubricated bearings are run under a light thrust load for 60 ± 3 s to distribute the lubricant in a pattern that might be found in service. The bearings are exposed to a solution of synthetic sea water at some percentages in distilled water, then stored for 24 ± 0.5 h at 52 ± 1 °C and 100 % relative humidity. After cleaning, the bearing cups are examined for evidence of corrosion.

TEST PRECISION

Traditional ways of expressing precision are not available. However, in a round robin study, 92 % of the duplicate results were in agreement. Similarly 77 % of the six labs matched results. This exercise involved six laboratories and eight samples at four concentrations of synthetic sea water (0 %, 5 %, 10 %, and 100 %).

Since this is a pass-fail test, no bias statement regarding this method can be made.

CORROSION-PREVENTIVE PROPERTIES OF LUBRICATING GREASES (EMCOR TEST):

D 6138

EXPLANATION

This test method is used to assess the ability of grease to prevent corrosion in rolling bearings operated in the presence of distilled water, NaCl solution, or synthetic sea water. It uses grease-lubricated ball bearings under dynamic wet conditions. It is used for development and specification purposes.

TEST SUMMARY

New, cleaned, and lubricated bearings are tested partially immersed in water (distilled, synthetic sea water, or NaCl solution) under no applied load at a speed of 83 ± 5 r/min in a predetermined sequence of running and stopping for a period of approximately one week. After cleaning, the bearings are examined and rated according to the degree of corrosion.

TEST PRECISION

Test Fluid	Repeatability	Reproducibility
Water, Reagent Grade	1	1
Synthetic Sea Water	1	2
NaCl Solution (0.5 mole/L)	1	2

This method has no bias because the value of corrosion can be defined only in terms of this test method.

CRYSTALLINE SIZE OF CALCINED PETROLEUM COKE BY X-RAY DIFFRACTION:

D 5187

EXPLANATION

Crystallinity of petroleum coke is a general measure of quality affecting suitability for end use and is a function of the heat treatment. The crystallite thickness is used to determine the extent of such heat treatment, for example, during calcinations. The value of L_c determined is not affected by coke microporosity or the presence of foreign, noncrystalline material such as dedust oil.

An X-ray diffraction pattern of a representative, pulverized sample of calcined petroleum coke is obtained using conventional X-ray scanning techniques. Calcined petroleum coke contains crystallites of different thickness of all crystallites in the sample by empirical interpretation of the X-ray diffraction pattern. The crystallite diameter L_a is not determined by this method.

TEST SUMMARY

A packed sample pulverized to less than 75 μm is subjected to a monochromatic X-ray beam and rotated to produce a diffraction pattern under specific conditions. The location and the shape of the peak with $hkl = (002)$ at $d = 3.35$ A^0 is used to calculate the L_c by manual interpretation of the peak or by computer simulation.

TEST PRECISION

Repeatability:	0.021 *X*
Reproducibility:	0.11 *X*

Where *X* is the average of two results in angstroms.

The bias of this test method has not been determined since there is no accepted reference material for this analysis.

This test method has no bias.

DEMULSIBILITY CHARACTERISTICS OF LUBRICATING OILS:
D 2711

EXPLANATION

This test method provides a guide for determining the demulsibility characteristics of lubricating oils that are prone to water contamination and that may encounter the turbulence of pumping and circulation capable of producing water-in-oil emulsions. This test method is intended for use in testing medium and high-viscosity lubricating oils.

TEST SUMMARY

A 405-mL sample of oil and 45 mL of distilled water are stirred together for 5 min at 180°F(82°C) in a special graduated separatory funnel. After a 5-h settling period the percentage of water in the oil, the volumes of water, and emulsion separating from the oil are measured and recorded.

A modification of this procedure for evaluating oils containing extreme pressure additives is also available. The modifications include volume of oil 360 mL, volume of distilled water 90 mL, and a stirring speed 2500 ± 250 r/min.

TEST PRECISION

Analysis	Repeatability	Reproducibility
Total free water, mL	4.0	8.0
Emulsion, mL	0.2	0.3

This test method has no bias.

DENSITY, RELATIVE DENSITY, AND SPECIFIC GRAVITY

GENERAL

Density is an important property of petroleum products being part of product specifications. Materials are usually bought and sold on that basis or if on volume basis then converted to mass basis via density measurements. This property is almost synonymously termed as density, relative density, gravity, and specific gravity, all terms related to each other. There are at least a dozen test method standards in DO2 manuals that describe this determination (see Table 8). Usually a hydrometer, pycnometer or a more modern digital density meter is used in all these standards. The plurality of the methods is because they are specifically written for different products. Some basic definitions:

Density—The mass (weight in vacuo) of liquid per unit volume at 15°C.

Relative density—The ratio of the mass of a given volume of liquid at 15°C to the mass of an equal volume of pure water at the same temperature.

Specific gravity—Same as relative density.

Real density is obtained when the particle size of the specimen is smaller than 75 μm. It directly influences the physical and chemical properties of the manufactured carbon and graphite artifacts for which it is used.

REAL DENSITY OF CALCINED PETROLEUM COKE:
D 2638

TEST SUMMARY

A sample is dried and ground to pass a 75-μm screen. The mass of the volume is determined directly, and the volume derived by the volume of helium displaced when the sample is introduced into a helium pycnometer. The ratio of the mass of the sample to the volume is reported as the real density.

TEST PRECISION

The test has a repeatability of 0.018 and reproducibility of 0.025 g/cm^3.

Bias of this test method is not known.

TABLE 8—*Comparison of Density Methods.*

Test Method	Property	Matrix	Technique	*r*	*R*	Bias
D 71	Relative Density	Solid Pitch	Gravimetry	0.005	0.007	NA[a]
D 1217	Density	Liquid Fuels	Bingham Pycnometer	0.00002	0.00003	NA
D 1298	Specific Gravity	Liquid Fuels	Hydrometer	Variable	Variable	NA
D 1480	Density and Relative Density	Viscous Liquids	Bingham Pycnometer	0.00005	0.00014	NA
D 1481	Relative Density	Viscous Liquids	Lipkin Pycnometer	0.00015	0.00035	NA
D 1657	Density	Light Hydrocarbons and LPG	Hydrometer	NA	NA	NA
D 2320	Density and Specific Gravity	Solid Pitch	Pycnometer	0.011(SG)	0.001(SG)	NA
				0.007(D)	0.011(D)	NA
D 2638	Real Density	Calcined Petroleum Coke	Helium Pycnometer	0.018	0.025	NA
D 4052	Density	Liquids	Digital Meter	0.0001	0.0005	0.0006
D 4292	Bulk Density	Calcined Petroleum Coke	Helium Pycnometer	0.014	0.046	NA
D 4892	Density	Solid Pitch	Pycnometer	0.02	0.03	0.04
D 5002	Specific Gravity	Crude Oil	Oscillating Frequency	0.00105X	0.00412X	NA
D 5004	Real Density	Calcined Petroleum Coke	Helium Pycnometer	0.0067	0.0156	NA

[a] NA—Not available.

DENSITY, RELATIVE DENSITY, AND SPECIFIC GRAVITY VIBRATED BULK DENSITY OF CALCINED PETROLEUM COKE:

D 4292

Vibrated bulk density (VBD) indicates the porosity of calcined petroleum coke, which affects its suitability for use in pitch-bonded carbon applications. This property is strongly dependent upon average particle size and range. VBD tends to increase with decreasing coke size. The procedure is limited to particles passing through a 6.68-mm opening sieve and retained on a 0.21-mm opening sieve.

TEST SUMMARY

After appropriate crushing the calcined coke with a jaw crusher and roll crusher, 100 g is measured after vibration and the bulk density is calculated.

TEST PRECISION

This test method has a repeatability of 0.014 and a reproducibility of 0.046 g/cm^3.

This test method is empirical and no statement as to bias can be made.

DENSITY AND RELATIVE DENSITY REAL DENSITY OF CALCINED PETROLEUM COKE:

D 5004

See Test Method D 2638 for discussion.

TEST SUMMARY

The mass of the sample is determined directly and the volume derived by determining the mass of liquid displaced when the sample is introduced into a pycnometer. Oil or other material sprayed on calcined petroleum coke to control dust will interfere. Such oil can be removed by flushing with a solvent, which also must be completely removed before the density determination.

TEST PRECISION

This test method has a repeatability of 0.0067 and a reproducibility of 0.0156.

Bias of this test method is not known.

DENSITY AND RELATIVE DENSITY GRAVITY, SPECIFIC: D 5002

EXPLANATION

This test method covers the determination of the density or relative density of crude oils that can be handled in a normal fashion as liquids at test temperatures between 15 and 35°C. This test method applies to crude oils with high vapor pressures provided appropriate precautions are taken to prevent vapor loss during transfer of the sample to the density analyzer.

TEST SUMMARY

Approximately 0.7 mL of crude oil sample is introduced into an oscillating sample tube and the change in oscillating frequency caused by the change in mass of the tube is used in conjunction with calibration data to determine the density of the sample.

TEST PRECISION

	Range	Limit
Repeatability:	0.75 to 0.95	$0.00105X$
Reproducibility:	0.75 to 0.95	$0.00412X$

Where X is sample mean.

DENSITY AND RELATIVE DENSITY BY DIGITAL METERS:

D 4052

(Equivalent Test Methods: IP 365, ISO 12185, DIN 51757D, JIS K 2249D, and AFNOR T60-172)

EXPLANATION

See under Gravity, API: Test Method D 287. This test method uses an automated digital density meter.

TEST SUMMARY

A small volume (approximately 0.7 mL) of liquid sample is introduced into an oscillating sample tube and the change in oscillating frequency caused by the change in the mass of the tube is used in conjunction with calibration data to determine the density of the sample.

This test method covers the determination of the density or relative density of petroleum distillates and viscous oils that can be handled in a normal fashion as liquids at test temperatures between 15 and 35°C. Its application is restricted to liquids with vapor pressures below 600 mm Hg (80 kPa) and viscosities below about 15 000 cSt(mm^2/s) at the temperature of test.

This test method should not be applied to samples so dark in color that the absence of air bubbles in the sample cell cannot be established with certainty. For the determination of density in crude oil samples use Test Method D 5002.

TEST PRECISION

	Range, g/mL	Limit
Repeatability:	0.68–0.97	0.0001
Reproducibiity:	0.68–0.97	0.0005
Bias:	0.68–0.97	0.0006

DENSITY AND RELATIVE DENSITY OF LIGHT HYDROCARBONS BY PRESSURE THERMOHYDROMETER:

D 1657

(Equivalent Test Methods: IP 235, ISO 3993, and AFNOR M41-008)

EXPLANATION

This test method covers the determination of the density or relative density of light hydrocarbons including liquid petroleum gas having Reid vapor pressure exceeding 101.325 kPa. The prescribed apparatus should not be used for materials having vapor pressures higher than 1.4 MPa at the test temperature.

TEST SUMMARY

The apparatus is purged with a portion of the sample before filling with the portion of the sample to be tested. The pressure cylinder is filled to a level at which the enclosed hydrometer floats freely. The hydrometer reading and the temperature of the sample are noted.

TEST PRECISION

Repeatability:	1 kg/m^3
Reproducibility:	3 kg/m^3

Bias of this test method has not been determined.

DENSITY AND RELATIVE DENSITY OF LIQUIDS BY BINGHAM PYCNOMETER:

D 1217

Originally this test method was developed for the determination of density of ASTM Knock Test Reference Fuels with an accuracy of 0.00003 g/mL. Although it is no longer used extensively for that purpose, the method is still useful whenever accurate densities of pure hydrocarbons or petroleum fractions with boiling points between 90 and 100°C are required.

TEST SUMMARY

The liquid sample is introduced into a pycnometer equilibrated to the desired temperature, and weighed. Weight of water required to fill the pycnometer at the same temperature is also measured. Both weights are corrected for the buoyancy of air before calculating density and relative density.

TEST PRECISION

This test method has a repeatability of 0.00002 and a reproducibility of 0.00003.

Specific bias has not been determined.

DENSITY AND RELATIVE DENSITY GRAVITY, SPECIFIC: D 1298

(Equivalent Test Methods: IP 160, ISO 3675, DIN 51757H, JIS K 2249H, and AFNOR T60-101)

EXPLANATION

See under Gravity, API Test Method D 287.

TEST SUMMARY

The sample is brought to the prescribed temperature and transferred to a cylinder at approximately the same temperature. The appropriate hydrometer is lowered into the sample and allowed to settle. After temperature equilibrium has been reached, the hydrometer scale is read, and the temperature of the sample is noted. If necessary the cylinder and its contents may be placed in a constant temperature bath to avoid excessive temperature variation during the test.

TEST PRECISION

Product	Temperature Range	Units	Repeatability	Reproducibility
Transparent	−2 to 24.5°C	Density	0.0005	0.0012
Nonviscous	29 to 76°F	Specific Gravity	0.0005	0.0012
	42 to 78°F	API Gravity	0.1	0.3
Opaque	−2 to 24.5°C	Density	0.006	0.0015
	29 to 76°F	Specific Gravity	0.0006	0.0015
	42 to 78°F	API Gravity	0.2	0.5

DENSITY AND RELATIVE DENSITY OF SOLID PITCH BY PYCNOMETER: D 2320

This test method can be used for the determination of density and specific gravity of pitch that can be handled in fragments.

TEST SUMMARY

The sample is weighed and its volume determined by the weight of water displaced in a pycnometer.

TEST PRECISION

Measurement	Repeatability	Reproducibility
Specific Gravity	0.011	0.001
Density	0.007 g/cm^3	0.011 g/cm^3

This test method has no bias.

DENSITY AND RELATIVE DENSITY OF SOLID PITCH BY PYCNOMETER:

D 4892

With this test method, density is determined to decimal places that are sufficient for most applications. A more precise measurement can be obtained using Test Methods D 71 or D 2320. This test method is applicable at a range of room temperatures of 15 to 35°C.

TEST SUMMARY

Bulk samples suspected of having free moisture should be air-dried or oven dried at 50°C before analyzing. The sample is pulverized and screened to a given fraction. The volume of a weighed portion of the fraction is determined using a helium pycnometer.

TEST PRECISION

This test method has a repeatability of 0.02 and a reproducibility of 0.03. The density determined by this test method was higher than that determined by water pycnometer method, Test Method D 2320 by an average of 0.004.

DENSITY AND RELATIVE DENSITY OF SOLID PITCH AND ASPHALT:

D 71

This test method determines the relative density by water displacement of hard pitches and asphalts with softening points above 70°C. Whenever possible homogenous natural fragments of sample free of cracks should be used. The use of cast cubes is not recommended due to the difficulty avoiding incorporation of air bubbles.

TEST SUMMARY

The sample is suspended from a thin wire and weighed, first in air, then submerged in water at 25°C. The relative density is calculated from these weights.

TEST PRECISION

This test method has a repeatability of 0.005 and a reproducibility of 0.007.

DYNAMIC VISCOSITY AND DENSITY BY STABINGER VISCOMETER:

D 7042

EXPLANATION

Density and viscosity are important properties of petroleum and nonpetroleum liquids. This test method provides concurrent measurements of both dynamic viscosity and density of liquid petroleum products and crude oils. The kinematic viscosity can be obtained by dividing the dynamic viscosity by density obtained at the same temperature. This test method is intended for liquids following Newtonian behavior. Although precision only in a specific range has been determined, the method should be applicable to a wider range of products.

TEST SUMMARY

The test specimen is introduced into the measuring cells, which are at a closely controlled and known temperature. The measuring cell consists of a pair of rotating concentric cylinders and an oscillating U-tube. The dynamic viscosity is determined from the equilibrium rotational speed of the inner cylinder under the influence of the shear stress of the test specimen and an eddy current brake in conjuction with adjustment data. The density is determined by oscillation frequency of the U-tube in conjuction with adjustment data.

The kinematic viscosity is calculated by dividing the dynamic viscosity by the density.

TEST PRECISION

The method is found not to have relative bias against D 445 kinematic viscosity method or D 4052 density method. Accepted reference materials were not tested by this method, hence no statement for bias can be made.

Repeatability (95 %). NOTE—X is the average of results being compared.

	15°C	40°C	100°C
Dynamic viscosity of base oils	not available	0.00101 X (0.10 %)	0.0003516 (X+5)
Kinematic viscosity of base oils	not available	0.00094 X (0.09 %)	0.0003473 (X+5)
Density of base oils	0.00046 g/mL	0.00030 g/mL	0.00033 g/mL

Reproducibility (95 %). NOTE—X is the average of results being compared.

	15°C	40°C	100°C
Dynamic viscosity of base oils	not available	0.00540 X (0.54 %)	0.002563 (X+5)
Kinematic viscosity of base oils	not available	0.00584 X (0.58 %)	0.002889 (X+5)
Density of base oils	0.00177 g/mL	0.00147 g/mL	0.00131 g/mL

DENSITY, RELATIVE DENSITY AND API GRAVITY BY THERMOHYDROMETER METHOD:
D 6822

EXPLANATION

Density and API gravity are used in custody transfer quantity calculations and to satisfy transportation, storage, and regulatory requirements. These are also the factors that indicate the quality of crude petroleum. Crude petroleum prices are often posted against values in kg/m^3 or in degrees API. However, this property of petroleum is an uncertain indication of its quality unless correlated with other properties.

Because the thermohydrometer incorporates both the hydrometer and thermometer in one device, it is more applicable in field operations for determining density or API gravity of crude petroleum and other liquid petroleum products with Reid vapor pressures of 101.325 kPa or less. This test method is suitable for analyzing low viscosity transparent or opaque liquids, or both. When used for opaque liquids, it requires the use of a meniscus correction.

TEST SUMMARY

The density or API gravity, after temperature equilibrium has been reached, is read by observing the freely floating thermohydrometer and noting the graduation nearest to the apparent intersection of the horizontal plane surface of the liquid with the vertical scale of the hydrometer after temperature equilibrium has been reached. The observed thermohydrometer reading is reduced to the reference temperature value by means of the Petroleum Measurement Tables, and observed temperature from the enclosed thermometer.

TEST PRECISION

Repeatability:	0.6 kg/m^3 or 0.2° API
Reproducibility	1.5 kg/m^3 or 0.5° API

Bias of this method has not been determined.

DENSITY AND RELATIVE DENSITY OF VISCOUS MATERIALS BY BINGHAM PYCNOMETER:
D 1480

This test method is applicable to liquids of vapor pressures below 600 mm Hg (80 kPa) and viscosities below 40 000 cSt at the test temperature. This test method can be used at temperatures between 20 and 100°C. It can be used at higher temperatures; however, in this case the stated precision of this test method does not apply.

For determination of density of materials, fluid at room temperature, use Test Method D 1217. This test method also provides a calculation procedure for converting density to specific gravity.

TEST SUMMARY

The liquid sample is introduced into the pycnometer, equilibrated to the desired temperature, and weighed. The density or relative density is then calculated from this weight and the previously determined calibration factor, and a correction is applied for the buoyancy of air.

TEST PRECISION

This test method has a repeatability of 0.00005 and a reproducibility of 0.00014 g/mL for a pycnometer volume of 10 mL. This precision statement may not apply if a pycnometer other than 10 mL in volume is used or if the temperature of test exceeds 100°C.

The difference of results from the established value when compared to pure reference materials is not expected to be more than ±0.00014 g/mL.

Specific bias has not been established for this test method.

DENSITY AND RELATIVE DENSITY OF VISCOUS MATERIALS BY LIPKIN PYCNOMETER:
D 1481

This test method is applicable to oils more viscous than 15 cSt at 20°C, and of viscous oils and melted waxes at elevated temperatures, but not at temperatures at which the sample would have a vapor pressure of 100 mm Hg (13 kPa) or above.

This test method provides a calculation for converting density to relative density (specific gravity). Densities of less viscosities at 20° or 25°C can be determined using Test Method D 1217.

TEST SUMMARY

The liquid is drawn into the bicapillary pycnometer through the removable siphon arm and adjusted to volume at the temperature of test, in such a manner that there is practically no drainage in the unfilled tubing. After equilibration at the test temperature, liquid levels are read, and the pycnometer is removed from the thermostated bath, cooled to room temperature, and weighed. From the weight of the sample and the volume at the test temperature density is calculated after correcting for the effect of air buoyancy.

TEST PRECISION

This test method has a repeatability of 0.00015 and a reproducibility of 0.00035 g/mL for a 10 mL volume pycnometer. This precision may not apply if a pycnometer of volume other than 10 mL is used.

The difference of results from established values when compared to pure reference materials is not expected to be more than 0.00035 g/mL.

Specific bias has not been established for this test method.

DEPENTANIZATION OF GASOLINE AND NAPTHAS: D 2001

EXPLANATION

The presence of pentane and lighter hydrocarbons in gasolines, napthas, and similar petroleum distillates interferes in the determination of hydrocarbon types by Test Methods D 1319 and D 2789. Pentane and lighter hydrocarbons are separated by this test method so that the depentanized residue can be analyzed, and pentane and lighter hydrocarbons can be analyzed by other methods, if desired. Typically about 2 volume % of C_5 and lighter hydrocarbons remain in the bottoms, and C_6 and heavier hydrocarbons carry over to the overhead.

TEST SUMMARY

A 50-mL sample is distilled into an overhead (C_5 and lighter) fraction and a bottoms (C_6 and heavier) fraction. The volume of bottoms is measured and the volume %, based on the charge, is calculated.

TEST PRECISION

This test method has a repeatability of 2 and a reproducibility of 4 volume %. This is an empirical test and a statement of bias is not appropriate.

DIESEL FUEL DILUENT IN USED DIESEL ENGINE OILS BY GAS CHROMATOGRAPHY: D 3524

EXPLANATION

Some fuel dilution of the engine oil may take place during normal operations. However, excessive fuel dilution is of concern in terms of possible performance problems. This test method covers the determination of diesel fuel as a contaminant in used lubricating oil. This test method is limited to SAE 30 oil. This test method may be applicable to higher viscosity grade oils. However, for such oils the precision statement of this test method may not be applicable. When testing unknown or mixed brands of used engine oil, the precision of the method may be poorer than the precision obtained when calibrating with a new oil representative of the used oil being tested.

TEST SUMMARY

A mixture of *n*-decane and used lubricating oil is introduced into a gas chromatographic column that separates hydrocarbons in the order of their boiling points. The column temperature is raised at a reproducible rate and the resulting chromatogram is interpreted for diesel fuel dilution.

TEST PRECISION

This test method has a repeatability of 0.3 mass % and a reproducibility of 1.6 mass %. This precision is based on the use of electronic integrators to measure areas and may not be indicative when other means of measurement are used.

Because of the empirical nature of this test, no estimate of bias for this test can be made.

DISTILLATION OF PETROLEUM PRODUCTS

GENERAL

The basic method of distillation to determine the boiling range of a petroleum product has been in use since the beginning of the petroleum industry. Distillation method, Test Method D 86 is one of the oldest methods under the jurisdiction of ASTM D02 Committee. Enormous amounts of data exist in the oil company archives using these distillation methods. The distillation characteristics of hydrocarbons have an important effect on their safety and performance, especially in the case of fuels and solvents. The boiling range gives information on the composition, the properties, and the behavior of fuels during storage and use. Volatility is the major determinant of the tendency of a hydrocarbon mixture to produce potentially explosive vapors. The distillation characteristics are critically important for both aviation and automotive gasolines, affecting starting, warm-up, and tendency to vaporlock at high operating temperatures or at high altitude, or both. The presence of high boiling components in these and other fuels can significantly affect the degree of formation of solid combustion deposits. Distillation limits are often included in petroleum product specifications, in commercial contract agreements, process refinery/control applications, and for regulatory compliance.

Several methods are described here for the distillation of various petroleum products. In addition to these physical methods, other test methods on gas chromatography can also give boiling point distribution (for example, Test Methods D 2887 and D 3710).

DISTILLATION OF CRUDE PETROLEUM: D 2892

(Equivalent Test Method: ISO 8708)

GENERAL

This test provides an estimate of the yields of fractions of various boiling ranges, and is therefore valuable in technical discussions of a commercial nature. This test method corresponds to the standard laboratory distillation efficiency referred to as 15/5. This test method can be used for any petroleum product except LPG, very light napthas, and fractions having initial boiling points above 400°C. This test method employs a fractionating column having an efficiency of 14 to 18 theoretical plates operated at a reflux ratio of 5:1.

TEST SUMMARY

A weighed sample of 1 to 30 L of stabilized crude petroleum is distilled to a maximum temperature of 400°C in a fractionating column having an efficiency at total reflux of at least 14 but not greater than 18 theoretical plates. A reflux ratio of 5 to 1 is maintained at all operating pressures. Temperature, pressure, and other variables are recorded at intervals and at the end of each cut or fraction. The mass and density of each fraction or cut are obtained. Distillation yields by mass are calculated from the mass of all fractions. Distillation yields by volume of all fractions are calculated from mass and density. From these data the TBP curves in mass or volume %, or both, versus atmospheric equivalent temperature are drawn.

TEST PRECISION

The repeatability of this test method is being reviewed. Following reproducibility can be obtained:

Atmospheric pressure	1.2 mass %	1.2 volume %
Vacuum pressure	1.4	1.5

Bias of this test method has not been determined.

DISTILLATION OF HEAVY HYDROCARBON MIXTURES: D 5236

This is a complex procedure involving many interacting variables. It is most important that at the time of first use of a new apparatus, its components be checked in detail as described in the method. This method describes a procedure for distilling heavy hydrocarbon mixtures having initial boiling points greater than 150°C(300°F), such as heavy crude oils, petroleum distillates, residues, and synthetic mixtures. It employs a potstill with a low pressure drop entrainment separator operated under total takeoff conditions. The maximum achievable temperature, up to 565°C (1050°F) is dependent upon the heat tolerance of the charge.

TEST SUMMARY

A weighed volume of the sample is distilled at absolute pressures between 6.6 and 0.013 kPa (50 and 0.1 mm Hg) at specified distillation rates. Cuts are taken at preselected temperatures. Records of vapor temperature, operating pressure, and other variables are made at intervals, including at each cut point. The mass and density of each fraction are obtained. From this the distillation yields by mass and volume are calculated.

TEST PRECISION

Liquid Volume % Distilled	Repeatability, °C	Reproducibility, °C
10	6.1	16.9
20	4.5	12.8
30	6.1	13.5
40	4.9	11.2
50	5.7	14.2
60	4.1	8.4
70	4.8	11.4
80	4.9	5.1
90	4.4	4.4

The bias of this test method has not been determined.

DISTILLATION OF PETROLEUM PRODUCTS:

D 86

(Equivalent Test Methods: IP 123, ISO 3405, DIN 51751, JIS K 2254, and AFNOR M07-002)

EXPLANATION

Distillation (volatility) properties of petroleum products are indicative of their performance. The empirical results obtained by this test method have been found to correlate with automotive equipment performance factors and with volatility characteristics of the sample.

This test method covers the distillation of motor and aviation gasolines, aviation turbine fuels, special boiling point spirits, naphthas, white spirit, kerosene, gas oils, distillate fuel oils, and similar petroleum products.

Test Methods D 86 and D 1078 differ only in heating rates and flask size used. Test Method D 86 is used for heavier fluids with a dry point >140°C.

DEFINITIONS OF TERMS USED

initial boiling point—the thermometer reading that is observed at the instant that the first drop of condensate falls from the lower end of the condenser tube.

end point or final boiling point—the maximum thermometer reading obtained during the test. This usually occurs after the evaporation of all liquid from the bottom of the flask. The term “maximum temperature” is a frequently used synonym.

dry point—the thermometer reading that is observed at the instant the last drop of liquid evaporates from the lowest point in the flask. Any drops of film of liquid on the side of the flask or on the thermometer are disregarded.

decomposition point—the thermometer reading that coincides with the first indications of thermal decomposition of the liquid in the flask. Characteristic indications of thermal decomposition are an evolution of fumes, and erratic thermometer readings, which usually show a decided decrease after any attempt is made to adjust the heat.

percent recovered—the volume in millilitres of condensate observed in the receiving graduate, in connection with a simultaneous thermometer reading.

percent recovery—the maximum percent recovered.

percent total recovery—the combined percent recovery and residue in the flask.

percent loss—100 minus the percent total recovery.

percent residue—the percent total recovery minus the percent recovery, or the volume of residue in millilitres if measured directly.

percent evaporated—the sum of the percent recovered and the percent loss.

TEST SUMMARY

A 100-mL sample is distilled (manually or automatically) under conditions prescribed, in the test method, appropriate to its nature. Thermometer readings and volumes of condensate are systematically recorded, and from these data, the results are calculated and reported as any or all of the above characteristics.

Repeatability and Reproducibility for Group 1.

Evaporated Point, %	Manual Repeatability[a]		Manual Reproducibility[a]		Automated Repeatability[a]		Automated Reproducibility[a]	
	°C	°F	°C	°F	°C	°F	°C	°F
IBP	3.3	6	5.6	10	3.9	7	7.2	13
5	$1.9+0.86S_C$	$3.4+0.86S_F$	$3.1+1.74S_C$	$5.6+1.74S_F$	$2.1+0.67S_C$	$3.8+0.67S_F$	$4.4+2.0S_C$	$7.9+2.0S_F$
10	$1.2+0.86S_C$	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$	$1.7+0.67S_C$	$3.0+0.67S_F$	$3.3+2.0S_C$	$6.0+2.0S_F$
20	$1.2+0.86S_C$	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$	$1.1+0.67S_C$	$2.0+0.67S_F$	$3.3+2.0S_C$	$6.0+2.0S_F$
30–70	$1.2+0.86S_C$	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$	$1.1+0.67S_C$	$2.0+0.67S_F$	$2.6+2.0S_C$	$4.7+2.0S_F$
80	$1.2+0.86S_C$	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$	$1.1+0.67S_C$	$2.0+0.67S_F$	$1.7+2.0S_C$	$3.0+2.0S_F$
90	$1.2+0.86S_C$	$2.2+0.86S_F$	$0.8+1.74S_C$	$1.4+1.74S_F$	$1.1+0.67S_C$	$2.0+0.67S_F$	$0.7+2.0S_C$	$1.2+2.0S_F$
95	$1.2+0.86S_C$	$2.2+0.86S_F$	$1.1+1.74S_C$	$1.9+1.74S_F$	$2.5+0.67S_C$	$4.5+0.67S_F$	$2.6+2.0S_C$	$4.7+2.0S_F$
FBP	3.9	7	7.2	13	4.4	8	8.9	16

[a] S_C or S_F is the average slope (or rate of change) calculated in accordance with 13.2.

Repeatability and Reproducibility for Groups 2, 3 and 4 (Manual Method).

	Repeatability[a]		Reproducibility[a]	
	°C	°F	°C	°F
IBP	$1.0+0.35S_C$	$1.9+0.35S_F$	$2.8+0.93S_C$	$5.0+0.93S_F$
5–95 %	$1.0+0.41S_C$	$1.8+0.41S_F$	$1.8+1.33S_C$	$3.3+1.33S_F$
FBP	$0.7+0.36S_C$	$1.3+0.36S_F$	$3.1+0.42S_C$	$5.7+0.42S_F$
% volume at temperature reading	$0.7+0.92/S_C$	$0.7+1.66/S_F$	$1.5+1.78/S_C$	$1.53+3.20/S_F$

[a] Calculate S_C or S_F from 13.2.

Repeatability and Reproducibility for Groups 2, 3 and 4 (Automated).

	Repeatability[a]		Reproducibility[a]	
Collected, %	°C	°F	°C	°F
IBP	3.5	6.3	8.5	15.3
2 %	3.5	6.3	$2.6+1.92S_C$	$4.7+1.92S_F$
5 %	$1.1+1.08S_C$	$2.0+1.08S_F$	$2.0+2.53S_C$	$3.6+2.53S_F$
10 %	$1.2+1.42S_C$	$2.2+1.42S_F$	$3.0+2.64S_C$	$5.4+2.64S_F$
20–70 %	$1.2+1.42S_C$	$2.2+1.42S_F$	$2.9+3.97S_C$	$5.2+3.97S_F$
80 %	$1.2+1.42S_C$	$2.2+1.42S_F$	$3.0+2.64S_C$	$5.4+2.64S_F$
90–95 %	$1.1+1.08S_C$	$2.0+1.08S_F$	$2.0+2.53S_C$	$3.6+2.53S_F$
FBP	3.5	6.3	10.5	18.9

[a] S_C or S_F is the average slope (or rate of change) calculated in accordance with 13.5.

DISTILLATION AT REDUCED PRESSURE:
D 1160
(Equivalent Test Method: ISO 6616)

This test method is used for the determination of the distillation characteristics of petroleum products and fractions that may decompose if distilled at atmospheric pressure. Both a manual and and an automatic method are included. The referee method is the manual method at mutually agreed upon pressure. Many engineering design correlations have been developed on data by this test method. These correlative methods have been used extensively in current engineering practice.

TEST SUMMARY

The sample is distilled at an accurately controlled pressure between 0.13 and 6.7 kPa (1 and 50 mm Hg) under conditions designed to provide approximately one theoretical plate fractionation. Data are obtained from which the initial and the final boiling points, and a distillation curve relating volume % distilled and atmospheric equivalent boiling point temperature can be prepared.

TEST PRECISION[1]

See the following table for the precision data obtained from a 1983 cooperative interlaboratory program. The bias of this method has not been determined.

Precision.

NOTE—The body of this table is in degrees Celsius atmospheric equivalent temperature.

Criteria Pressure	Repeatability				Reproducibility			
	0.13 kPa(1 mm Hg)		0.13 kPa(10 mm Hg)		0.13 kPa(1 mm Hg)		1.3 kPa(10 mm Hg)	
IBP	17		15		56		49	
FBP	3.3		7.1		31		27	
Volume Recovered	5–50 %	60–90 %	5–50 %	60–90 %	5–50 %	60–90 %	5–50 %	60–90 %
C/V %								
0.5	2.4	2.5	1.9	2.0	6.5	3.9	7.0	5.4
1.0	2.9	3.0	2.4	2.5	10	6.0	9.3	7.2
1.5	3.2	3.3	2.8	2.9	13	7.8	11	8.5
2.0	3.4	3.5	3.1	3.2	16	9.4	12	9.6
2.5	3.6	3.7	3.3	3.5	18	11	14	11
3.0	3.8	3.9	3.6	3.7	21	12	15	11
3.5	3.9	4.0	3.8	3.9	23	13	16	12
4.0	4.0	4.2	3.9	4.1	25	15	16	13
4.5	4.1	4.3	4.1	4.3	27	16	17	13
5.0	4.2	4.4	4.3	4.4	29	17	18	14
5.5	4.3	4.5	4.4	4.6	30	18	19	15
6.0	4.4	4.6	4.5	4.7	32	19	19	15
6.5	4.5	4.7	4.7	4.8	34	20	20	16
7.0	4.6	4.8	4.8	5.0	35	23	21	16
7.5	4.7	4.8	4.9	5.1	37	22	21	16
8.0	4.8	4.9	5.0	5.2	38	23	22	17
8.5	4.8	5.0	5.1	5.3	40	24	22	17
9.0	4.9	5.1	5.2	5.4	41	25	23	18
9.5	5.0	5.1	5.3	5.5	43	25	23	18
10.0	5.0	5.2	5.4	5.6	44	26	24	19
10.5	5.1	5.2	5.5	5.7	46	27	24	19
11.0	5.1	5.3	5.6	5.8	47	28	25	19
11.5	5.2	5.4	5.7	5.9	48	29	25	20
12.0	5.2	5.4	5.8	6.0	50	30	26	20
12.5	5.3	5.5	5.9	6.1	51	30	26	20
13.0	5.3	5.5	6.0	6.2	52	31	27	21
13.5	5.4	5.6	6.0	6.3	54	32	27	21
14.0	5.4	5.6	6.1	6.3	55	33	27	21
14.5	5.5	5.7	6.2	6.4	56	33	28	22
15.0	5.5	5.7	6.3	6.5	57	34	28	22

[1] This table is Table 7 in Test Method D 1160.

DISTILLATION OF PITCH:
D 2569

This test method for the distillation of pitch can be used when the amount of distillate below 270°C is <2 %.

TEST SUMMARY

A 100-g sample is distilled from an electrically heated flask in a shield at a designated rate. The weight of distillate fractions at specified temperatures and the time required to collect the main fraction are recorded

TEST PRECISION

This method has a repeatability of 1.5 and a reproducibility of 3.0 percentage points for the total distillate to 360°C.

This test method has no bias.

DIMETHYLFORMAMIDE OF TAR AND PITCH:
D 2764

EXPLANATION

This test method is used in evaluating and characterizing tars and pitches and as one element in establishing the uniformity of shipments or sources of supply. It is a rapid and reasonably accurate measure of the toluene insoluble (TI) content of tar and pitch Test Method D 4072. In this method dimethylformamide (DMF) insoluble matter in tar and pitch is determined. Since this test method is empirical, strict adherence to all details of the procedure is necessary.

TEST SUMMARY

A sample is digested in hot DMF and filtered. Any insoluble matter is washed, dried, and weighed.

TEST PRECISION

Repeatability:	0.4 %
Reproducibility:	0.8 %

This test method has no bias because the value of DMF insolubles is defined in terms of this test method.

DROPPING POINT

GENERAL

The dropping point is the temperature at which the grease passes from a semisolid to a liquid state. This change in state is typical of greases containing soaps of conventional types added as thickeners. These tests are useful in identifying the types of greases and for establishing and maintaining benchmarks for quality control.

There are two test methods for this purpose. In general, dropping points obtained by Test Methods D 566 and D 2265 are in agreement up to 260°C. In the case where results differ, there is no known significance.

DROPPING POINT OF LUBRICATING GREASE:
D 566
(Equivalent Tests: IP 132 and ISO 2176)

TEST SUMMARY

This test method is not recommended for temperatures above 288°C. In those cases, use Test Method D 2265. A sample of lubricating grease contained in a cup suspended in a test tube is heated in an oil bath at a prescribed rate. The temperature at which the material falls from the hole in the bottom of the cup is averaged with the temperature of the oil bath and recorded as the dropping point.

TEST PRECISION

This test method has a repeatability of 7°C and a reproducibility of 13°C.

This test method has no known bias.

DROPPING POINT OF LUBRICATING GREASE: D 2265

TEST SUMMARY

A grease sample in a test cup is supported in a test tube placed in an aluminum block oven at a preset constant temperature. A thermometer is positioned in the cup so that it measures the temperature without coming in contact with the grease. As the temperature increases, a drop of material will fall from the cup to the bottom of the test tube. Temperatures of both the thermometer and that of the block oven are recorded at this point. One third the difference between the two values is the correction factor which is added to the observed value, and reported as the dropping point of the grease.

TEST PRECISION

The following precision can be expected.

Dropping Point, °C	Repeatability	Reproducibility
Up to 116	6	9
116 up to 221	8	12
221 up to 277	6	16
227 up to 316	7	12

This test method has no bias.

DUST CONTROL MATERIAL ON CALCINED PETROLEUM COKE: D 4930

EXPLANATION

The dust control material is applied to calcined coke to help maintain a dust free environment. It adds weight to the coke and can have a negative effect on the quality of carbon and graphite artifacts made from the treated coke. Hence, the coke customer is interested in the amount of it in coke, and may specify a maximum level. This test method is limited to those materials that are soluble in a solvent (for example, methylene chloride) that can be used in a Soxhlet extraction type of apparatus. Toluene and methyl chloroform have also been found to give equal results as methylene chloride.

TEST SUMMARY

From a weighed dry representative sample of 6.3 mm of maximum sized coke, the dust control material is dissolved by methylene chloride in a Soxhlet apparatus. The mass of the residue remaining after extraction and evaporation of the solvent is the mass of the dust control material.

TEST PRECISION

The precision of this test is dependent on the absolute amount of oil on the coke as well as the efficiency of sampling and riffling procedure used. A repeatability of 0.04 and a reproducibility of 0.15 has been found.

This is an empirical test and no statement about bias can be made.

ELASTOMER COMPATIBILITY OF LUBRICATING GREASES AND FLUIDS: D 4289

EXPLANATION

Measurement of changes in the volume and hardness of an elastomer that occur from immersion in a lubricating grease or fluid provide a means of evaluating relative compatibility. Much of this test method is derived from Test Method D 471 and Federal Test Method 791C/3603.5. Although this test method was specifically developed for evaluating greases used in automotive applications, it can also be applied to lubricants used in industrial applications. The volume and hardness-change values determined by this test method do not duplicate similar changes that occur in elastomeric seals under actual service conditions. However, they can be correlated in many instances.

Some lubricant specifications may require different test conditions, such as longer durations or lower or higher temperatures. In such cases, the precision values stated in this method do not apply. This test method can also be used to evaluate the compatibility of greases with rubber products not in standard sheet form.

TEST SUMMARY

A prepared elastomer coupon is measured for initial hardness (Durometer A), density or relative density, and volume by water displacement. The coupon is immersed in test grease or fluid and aged for 70 hours at either 100°C (for chloroprene and similar flex-resistant elastomers) or at 150°C (for nitrile and similar heat resistant elastomers) or at other conditions required by the lubricant specifications. The resulting change in hardness and volume is determined.

Because of possible degradation, in order to ensure acceptable precision in this test method, neither test greases nor test elastomers should be more than approximately one year old. If necessary, precautions should be taken to maintain integrity of the elastomers by storing under special conditions described in the test method.

TEST PRECISION

Reference Elastomer	Repeatability	Reproducibility
AMS 3217/2B (NBR-L) @ 150°C		
Volume Change	0.84	3.45
Hardness Change	1.51	11.5
AMS 3217/3A (CR) @ 100°C		
Volume Change	1.41	9.87
Hardness Change	1.5	4.5

Where volume change is in units of percentage points, and hardness change is in units of Durometer A units.

This test method has no bias because the values for volume change and hardness change can be defined only in terms of the test method.

ELECTRICAL CONDUCTIVITY OF FUELS: **D 2624** (Equivalent Tests: IP 274, ISO 6297, and DIN 51412 T2)

EXPLANATION

The ability of a fuel to dissipate charge that has been generated during pumping and filtering operations is controlled by its electrical conductivity, which depends upon its ionic species content. If the conductivity is high enough, charges dissipate fast to prevent their accumulation and avoid dangerously high potentials in a receiving tank. These test methods cover the determination of the electrical conductivity of aviation and distillate fuels with and without a static dissipator additive.

TEST SUMMARY

Two test methods are available for field tests of fuel conductivity: portable meters for direct measurements in tanks or the field or the laboratory measurements of fuel samples, and in-line meters for continuous measurement of fuel conductivity in a fuel distribution system. For specification purposes, conductivity measurements should be made with the portable meters.

A voltage is applied across two electrodes in the fuel and the resulting current is expressed as a conductivity value. With the portable meters, the current measurement is made almost instantaneously upon application of the voltage. This test method can measure conductivities from 1 to 2000 pS/m.

Precision[a] *of Emcee Models 1150, 1151, 1152, and 1153 and Maihak Indicator.*

Conductivity, pS/m	Repeatability	Reproducibility
1	1	1
15	6	3
20	7	4
30	9	6
50	13	10
70	15	13
100	19	17
200	29	32
300	37	45
500	51	69
700	62	92
1000	77	125
1500	98	177

[a] The precision limits in Table 1 are applicable at room temperatures; significantly higher precision (×2) may be applicable at temperatures near −20°C.

Precision[a] *of Maihak MLA 900 Meter.*

Conductivity, pS/m	Repeatability	Reproducibility
1	0	0
15	2	2
20	2	2
30	3	3
50	5	5
70	7	7
100	9	9
200	17	16
300	23	22
500	36	34
700	47	46
1000	64	61
1500	89	86

[a] The precision limits in Table 2 are applicable at room temperature; significantly higher precision (×2) may be applicable at temperatures near −20°C.

Test Method D 4308 should be used for measurements below 1 pS/m.

TEST PRECISION

See the following tables for precision data. The repeatability data are for both methods; however, the reproducibility estimates are only for the portable meter method.

The bias of this test method has not been determined.

ELECTRICAL CONDUCTIVITY OF LIQUID HYDROCARBONS:

D 4308

EXPLANATION

See discussion under Test Method D 2624. This test method applies to the determination of the “rest” conductivity of aviation fuels and other similar low-conductivity hydrocarbon liquids in the range 0.1 to 2000 pS/m. This test method can be used in the field or in a laboratory.

TEST SUMMARY

A sample of liquid hydrocarbon is introduced into a clean conductivity cell which is connected in series to a battery voltage source and a sensitive dc ammeter. The conductivity, automatically calculated from the observed peak current reading dc voltage and cell constant using Ohm's law, appears as a digital value in either a manual or automatic mode of meter operation.

TEST PRECISION

The precision statement was developed on samples in the range 0.1 to 2000 pS/m in an auto mode instrument (see Fig. 7). The bias of this test method has not been measured.

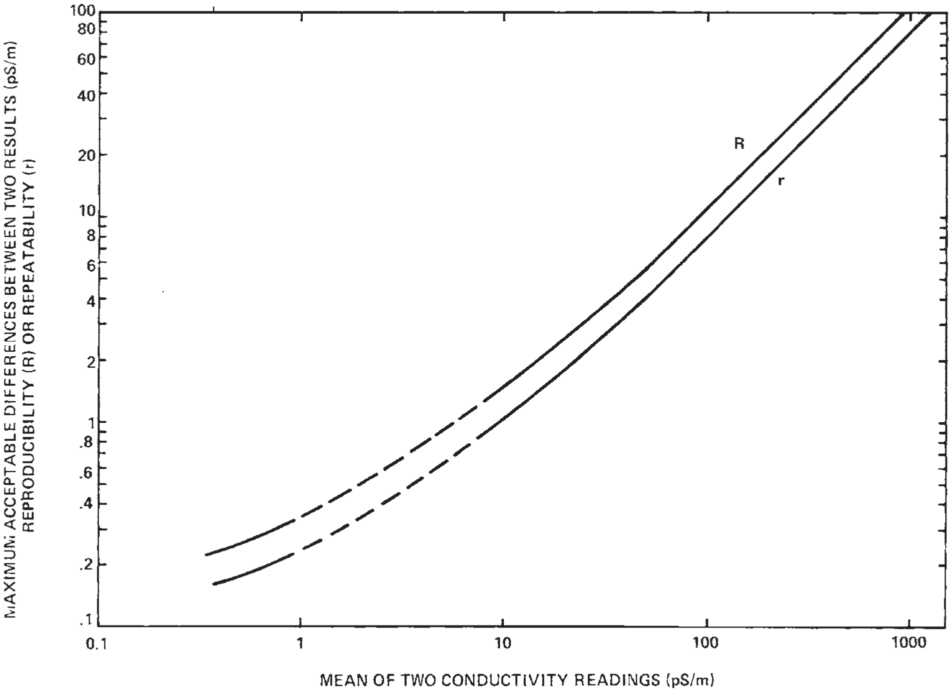


FIG. 7—*Variation of repeatability and reproducibility with conductivity level.*

ENGINE OIL VOLATILITY BY CAPILLARY GAS CHROMATOGRAPHY:

D 6417

EXPLANATION

The determination of engine oil volatility at 371°C is a requirement in some lubricant specifications. This test method is intended as an alternative to Test Methods D 5800 and D 5480. The data obtained by this test method are not directly equivalent to that obtained by Test Method D 5800. The results by this test method can be biased by the presence of additives (polymeric materials), or by heavier base oils which may not completely elute from the gas chromatographic column. The results of this test method may also not correlate with other oil volatility methods for nonhydrocarbon synthetic oils. This test method can be used on lubricant products not within the scope of other test methods using simulated distillation methodologies, such as Test Method D 2887.

This test method applicability is limited to samples having an initial boiling point greater than 126°C. This test method may be applied to both lubricant oil base stocks and finished lubricants containing additive packages. However, because of the nonelution of heavier components of these additive packages, the results by this test method are biased low compared to those by the Test Method D 5480, which uses an internal standard to compensate for noneluted material.

TEST SUMMARY

A sample aliquot diluted with a viscosity reducing solvent is introduced into the gas chromatographic system which uses a nonpolar open tubular capillary gas chromatographic column for eluting the hydrocarbon components of the sample in the order of increasing boiling point. The column oven temperature is raised at a reproducible linear rate effect separation of the hydrocarbons. The quantitation is achieved with a flame ionization detector. The sample retention times are compared to those of known hydrocarbon mixtures, and the cumulative corrected area of the sample determined to the 371°C retention time is used to calculate the percentage of oil volatilized at 371°C.

TEST PRECISION

Repeatability:	$0.1352\ X^{0.5}$
Reproducibiity:	$0.6036\ X^{0.5}$

Where X is the volatility level.

Bias has not been determined.

ETHANOL CONTENT IN DENATURED FUEL ETHANOL BY GC:
D 5501

EXPLANATION

Fuel ethanol is required to be denatured with gasoline in accordance with Specification D 4806. State and federal laws specify the concentration of ethanol in gasoline blends. Hence, the determination of the amount of denaturant is important. This test method covers the determination of ethanol content of denatured fuel ethanol by gas chromatography. Ethanol is determined from 93 to 97 mass %, and methanol from 0.1 to 0.6 mass %. This test method does not identify and quantify methanol but does purport to identify all individual components that make up the denaturant. Water cannot be measured by this test method, but can be measured by procedure such as Test Method D 1364. The result would be used to correct the chromatographic values. This method is inappropriate for impurities that boil at temperatures above 225°C, or for impurities that cause poor or no response in a flame ionization detector, such as water.

TEST SUMMARY

A fuel ethanol sample is injected into a gas chromatograph equipped with a methyl silicone bonded phase fused silica capillary column. Helium carrier gas transports the vaporized aliquot through the column where the components are separated. They are detected by a flame ionization detector as they elute from the column. The detector signal is processed by an electronic data acquisition system. The identification of all components is done by comparing their retention times with those of standards, and concentration is calculated by using normalized peak areas.

TEST PRECISION

Component	Range	Repeatability	Reproducibility
Ethanol	93–97	0.21	0.53
Methanol	0.01–0.6	$0.01859 \times \sqrt{X}$	$0.01172 \times \sqrt{X}$

Where X is the mass percent. The range is given in mass percent.

No significant difference was found between the ethanol or methanol content obtained by this test method and the expected content based on their concentration added to the samples.

ETHYL MERCAPTAN IN LPG VAPOR:
D 5305

EXPLANATION

LPG is colorless and odorless, and not detectable by normal human senses. To provide an olfactory warning in the event of a leak, LPG intended for domestic or commercial use is intentionally odorized so as to be readily detectable well below flammable or suffocating concentration levels of LPG in air. The most common odorant used is ethyl mercaptan.

This test method covers a rapid and simple procedure using length of stain tubes for field measurement of ethyl mercaptan in the vapor phase of LPG systems. Although length-of-stain tubes are available to detect ethyl mercaptan concentrations in the range of 0.5 to 120 ppm by volume, this test method is specifically applicable to systems containing 5 ppmv or more of ethyl mercaptan in LPG vapors. A chromatographic technique can be used for more precise, quantitative determination of ethyl mercaptan in LPG.

TEST SUMMARY

Using a manually operated vacuum pump, a sample of LPG is drawn through a detector tube made specifically for mercaptan detection. The length of stain (color change) produced in the detector tube when exposed to a measured volume of sample is directly proportional to the amount of ethyl mercaptan present in the sample being tested. The length of stain produced in the detector tube is converted to concentration, in ppmv, by comparison with a calibration scale provided by the stain tube manufacturer.

Detector tubes can be subject to interferences from materials other than the target substances. Methyl mercaptan will likely interfere; however, because different detection chemistry is used by different manufacturers, interferences can vary. Propylene will cause an interfering gray discoloration with some tubes.

TEST PRECISION

Concentration	Repeatability	Reproducibility
5 to 20 ppmv	15 %	20 %
Above 20 ppmv	20 %	20 %

Within the precision limits given above, this test method has no bias.

ETHYLENE GLYCOL IN USED ENGINE OIL:

D 4291

EXPLANATION

Leakage of aqueous engine coolant into the crank case weakens the ability of the oil to lubricate. If ethylene glycol is present, it promotes varnish and deposit formation. This test method provides for early detection to prevent coolant from accumulating and seriously damaging the engine. This test method is valid in the range 5 to 200 mass ppm of ethylene glycol.

Another procedure, Test Method D 2982, is also used for the determination of glycol antifreeze in used lube oils.

TEST SUMMARY

The sample of oil is extracted with water and the analysis is performed on the water extract which is injected into a gas chromatograph using on-column injection. The eluting compounds are detected by a flame ionization detector. The ethylene glycol peak area is determined and compared with areas obtained from injecting freshly prepared known standards.

TEST PRECISION

This test method has a repeatability of $0.212X$ and a reproducibility of $0.528X$ where X is ethylene glycol content in mass ppm.

The bias of this test method has not been determined.

EVAPORATION LOSSES BY VOLATILITY:

GENERAL

A number of methods exist for the determination of engine oil volatility based on gas chromatographic or physical measurements. Evaporation loss is a critical parameter of interest to automobile manufacturers, and is included in several oil lubricant specifications.

ENGINE OIL VOLATILITY BY GC:

D 5480

This test method provides the determination of the amount of engine oil volatilized at 700°F(371°C). This test method is applicable to engine oils containing high molecular weight components that need not elute from a GC column. This test method can also be used to determine the amount of oil volatilized at any temperature between 238 and 371°C, if so desired. This test method is limited to samples with an initial boiling point greater than 460°F(238°C). This test gives a more precise measurement of oil volatility than that obtainable using the traditional Noack method. This test method can also be used for lubricants not within the scope of Test Method D 2887.

TEST SUMMARY

The sample is mixed with an internal standard and a dilute tetracosane solution, and injected into a gas chromatograph. The column temperature is raised at a reproducible rate and the area under the chromatogram is recorded throughout the run. The retention time (*RT*) at 700°F is calculated using linear regression, utilizing the *n*-paraffins comprising the internal standard, and the tetracosane *RT*/boiling point data. The area response of the internal standard is related to the area of the sample determined to the 371°C*RT* to obtain the mass percentage of oil volatilized at 371°C.

TEST PRECISION

This test method has a repeatability of $0.403 \times X^{0.25}$ and a reproducibility of $1.671 \times X^{0.25}$, where X is the volatility level.

EVAPORATION LOSS OF LUBRICATING GREASES: D 2595

EXPLANATION

The loss of volatile materials from greases and oils can adversely affect the original performance characteristics of a lubricant and therefore, could be a significant factor in evaluating a lubricant for a specific use. Such volatiles can also become contaminants in the environment in which the lubricant is to be used. Correlation between results from this test method, and service performance has not been established.

This test method covers the temperature range of 93 to 316°C (200 and 600°F). This test augments Test Method D 972 which is limited to 149°C(300°F). This test method should not be used at temperatures that exceed the flash point of the base oil of the grease.

TEST SUMMARY

A weighed sample of grease in an evaporation cell is placed in a heating device and maintained at the desired test temperature. Heated air is passed over the grease surface for 22 ± 0.1 h. The loss in weight of the sample due to evaporation is then determined.

TEST PRECISION

This test method has a repeatability of 10 and a reproducibility of 15 % of the mean value.

This test method has no bias.

EVAPORATION LOSSES OF LUBRICATING GREASES AND OILS: D 972

EXPLANATION

See the discussion under Test Method D 2595. This test is run at temperatures between 100 and 150°C(210 to 300°F).

TEST SUMMARY

See the discussion under Test Method D 2595.

TEST PRECISION

This test method has a repeatability of 0.025 M and a reproducibility of 0.10 M, where M is the mean of two values.

This test method has no bias.

EVAPORATION LOSSES NOACK EVAPORATION LOSS: D 5800

(Equivalent Test Methods: IP 421, DIN 51581, and CEC-L14-A88)

EXPLANATION

With the introduction of a maximum volatility limit in European crankcase lubricant specifications there was a requirement for a volatility test with acceptable precision limits, since volatility limits may have a significant impact on the formulation and ultimate production costs. This test method is based on the principle of loss of mass at constant temperature under a constant stream of air. Standardization of the equipment is necessary to maintain the required precision and is obtained by checking the value with a datum oil of known value. This test method described, relates to one set of operating conditions but may be readily adapted to other conditions if required.

Procedure A is the original DIN and CEC method which uses Wood's metal as a heating medium. Procedure B is the automated ISL method which uses a non-Wood's metal heating method. Procedure C called Selby Noack method also does not use the Wood's metal. It can also collect the evolved gases for separate analysis if desired. Noack results determined using Procedures A and B show consistent differences. Procedure A gives slightly lower results versus Procedure B on formulated engine oils, while Procedure A gives higher results versus procedure B on basestocks. Interlaboratory tests have shown that Procedures A, B, and C yield essentially equivalent results with a correlation coefficient R^2 of 0.996.

TEST SUMMARY

The evaporation loss is defined as that mass of oil lost when the sample is heated in a test crucible, through which a constant flow of air is drawn.

A measured quantity of sample is placed in an evaporation crucible which is then heated to 250°C with a constant flow of air drawn through it for 60 min. The loss in mass of the oil is determined. The reproducibility of this test method is dependent on a mandatory use of standard apparatus and procedure.

TEST PRECISION

	Repeatability	Reproducibility
Procedure A (CEC)	5.8 %	18.3 %
Procedure B (ISL)	$0.095\ X^{0.5}$	$0.26\ X^{0.5}$
Procedure C (Selby)	0.81 %	1.62 %

Several companies have found a correlation of 21.5 % Noack volatility to be equivalent to 17 % volatility by GC-SD methods (see Test Methods D 2887 or D 5480).

NOTES

Attempts have been made to correlate Noack results with thermogravimetry, high temperature-GCD, and MCRT. See oil volatility GCD Test Method D 5480 and TGA volatility Test Method D 6375.

SAFETY

A great deal of concern has been expressed both in ASTM and IP regarding the use and handling of hazardous material used in the Noack test. The safety concerns are two-fold: (*a*) volatilization of heated oil, and (*b*) release of lead, tin, bismuth, and cadmium from the Wood's metal heating block at 250°C during testing. These are highly toxic, chronic poisons. An incident in Europe showed contamination of laboratory benches around the Noack analyzers with these metals. The analyzers *must* be operated in well-ventilated hoods. Further specific precautions may be necessary to prevent exposure to these toxic metal fumes and oil vapors. Savant Laboratory and ISL are now selling commercial instruments which do not use the Wood's metal.

EVAPORATION LOSSES BY THERMO GRAVIMETRIC ANALYSIS (TGA) NOACK METHOD:

D 6375

This is a safe and fast alternative for the determination of Noack evaporation loss of a lubricant. This test method is applicable to base stocks and fully formulated lubricating oils having a Noack evaporative loss from 0 to 30 mass %. This procedure requires a much smaller sample size. The evaporative loss determined by this test method is the same as that from the standard Noack method.

TEST SUMMARY

A lubricant sample is placed in an appropriate TGA specimen pan that is placed on the TGA pan holder and quickly heated to between 247 and 249°C under a stream of air, and then held isothermal for an appropriate time. Throughout this process, the TGA monitors and records the mass loss experienced by the specimen due to evaporation. The Noack evaporation loss is subsequently determined from the specimen's TG curve versus the Noack reference time determined under the same TGA conditions.

TEST PRECISION

This test method has a repeatability of $0.31X^{0.60}$ and a reproducibility of $0.39X^{0.60}$, where X is the TGA Noack volatility.

No significant bias was found between the Noack volatility determined by this test method and that determined by Test Method D 5800, Noack Method, within the repeatability of the test method.

EXPLOSIVE REACTIVITY OF LUBRICANTS:
D 3115

EXPLANATION

Explosive reactivity has resulted when parts made from some light alloys, typically high in aluminum and magnesium, are loaded under shear conditions while in contact with certain lubricants. A typical example is a threaded part, lubricated with a chlorofluorocarbon grease, pulled up normally tight. This test method is used to evaluate the explosive reactivity of lubricants in the presence of aerospace alloys under high shear conditions. The results of this test may be used as elements of a fire-hazard assessment which takes into account all other factors pertinent to a fire hazard.

TEST SUMMARY

A shaped dowel pin made of the test material is rotated at 1760 r/min under a pressure of 689 MPa(1000 psi) for 1 min in a shaped hole (drilled into a block of the test material) containing the test lubricant. Observation for indications of reaction is made.

TEST PRECISION

This test method is expected to have a repeatability not to exceed more than one reaction per set of six runs. The reproducibility should not exceed a difference of 108 % of the mean number of reactions.

EXTREME PRESSURE PROPERTIES OF FLUID LUBRICANTS:
D 3233

EXPLANATION

These test methods cover two procedures for making a preliminary evaluation of the load-carrying properties of fluid lubricants by means of the Falex Pin and Vee Block test machines. Evaluations by both test methods differentiate between fluids having low, medium, and high levels of extreme-pressure properties. The user should establish any correlation between results by either method and service performance.

Relative ratings by both test methods were in good agreement with four ball weld-point relative ratings obtained in Test Method D 2783.

TEST SUMMARY

Both test methods consist of running a rotating steel journal at 290 ± 10 r/min against two stationary *V*-blocks immersed in the lubricant sample. Load is applied to the *V*-blocks by a ratchet mechanism. In Test Method A (that is, Falex Run-up test) increasing load is applied continuously. In Test Method B (that is, Falex one-min step test) load is applied in 250 lbf increments with load maintained constant for 1 min at each load increment. In both these methods the load-fail value obtained is the criteria for level of load-carrying properties. Both methods require calibration of the load gage and reporting of test results as true (corrected) loads rather than actual gage loads.

TEST PRECISION

Test Method	Repeatability	Reproducibility
A	27% of the mean	40% of the mean
B	24 % of the mean	43 % of the mean

The procedure in these test methods has no bias.

EXTREME PRESSURE PROPERTIES OF LUBRICATING FLUIDS:

D 2782

(Equivalent Test Methods: IP 240 and DIN 51434)

EXPLANATION

This test method is widely used for the determination of extreme pressure properties for specification purposes of the load carrying capacity of lubricating fluids by means of Timken Extreme Pressure Tester. This test method is suitable for testing fluids having a viscosity of less than 5000 cSt at 40°C.

TEST SUMMARY

The test is operated with a steel test cup rotating against a steel test block at a rotating speed of 123.71 ± 0.77 m/min, which is equivalent to a spindle speed of 800 ± 5 r/min. Fluid samples are preheated to 37.8 ± 2.8°C before starting the test. Two determinations are made:

(*a*) The minimum load (score value) that will rupture the lubricant film being tested between the rotating cup and the stationary block and cause scoring or seizure; and

(*b*) The maximum load (OK value) at which the rotating cup will not rupture the lubricant film or cause scoring or seizure between the rotating cup and the stationary block.

TEST PRECISION

This test method has a repeatability of 30 and a reproducibility of 74 % of the mean value.

EXTREME PRESSURE PROPERTIES OF LUBRICATING FLUIDS:

D 2783

EXPLANATION

This test method is used for specification purposes, differentiates between lubricating fluids having low, medium, and high levels of extreme pressure properties. Two determinations are made: load-wear index, and weld point by means of four-ball extreme pressure tester.

TEST SUMMARY

The tester is operated with one steel ball under load rotating against three steel balls held stationary in the form of a cradle. Test lubricant covers the lower three balls. The rotating speed is 1760 ± 40 r/min. The machine and the lubricant are brought to 65–95°F and then a series of tests of 10-s duration are made at increasing loads until welding occurs. Ten tests are made below the welding point.

TEST PRECISION

This test method has a repeatability of 17 and a reproducibility of 44 % of the mean value for load wear index, and both repeatability and reproducibility of more than one increment loading for weld point measurement.

The bias of this test method is not known.

EXTREME PRESSURE PROPERTIES OF LUBRICATING GREASE: **D 2596**

EXPLANATION

See the discussion under Test Method D 2783. This test is not applicable to lubricating greases that have as their fluid component a silicone, halogenated silicone, or a mixture comprising silicone fluid and petroleum oil.

TEST SUMMARY

The tester is operated with one steel ball under load rotating against three steel balls held in the form of a cradle. The rotating speed is 170 ± 60 r/min. Lubricating greases are brought to a temperature of $27 \pm 8°F$ and then subjected to a series of tests of 10-s duration at increasing loads until welding occurs.

TEST PRECISION

Analysis	Repeatability	Reproducibility
Load Wear Index	0.14 X	0.44 X
Weld Point	One increment loading	One increment loading
Last Non-seizure Load	0.35 X	0.78 X

Where X is the average of two test results.

This procedure has no bias.

EXTREME PRESSURE PROPERTIES OF LUBRICATING GREASES:

D 5706

EXPLANATION

This test method is used for quickly determining extreme pressure properties of lubricating greases at selected temperatures specified for use in applications where high speed vibrational or start-stop motions are present with high Hertzian point contact. This test method has found wide application in qualifying lubricating greases used in constant velocity joints of front-wheel-drive automobiles.

TEST SUMMARY

This test method is performed on an SRV test machine using a steel test ball oscillating against a steel test disk with lubricant between them. Test load is increased in 100-N increments until seizure occurs. The load, immediately prior to the load at which seizure occurs, is measured and reported. Test frequency, stroke length, temperature, and ball and disk material can be varied to simulate field conditions.

TEST PRECISION

Conditions	Repeatability	Reproducibility
Tests run at 50°C	0.7 X	1.3 X
Tests run at 80°C	$0.6(X+122)$	$1.2(X+122)$

Where X is the average of two results.

This test method has no bias.

EXTREME PRESSURE PROPERTIES OF SOLID BONDED FILMS USING AN SRV TEST MACHINE:
D 7217

EXPLANATION

This test method covers a procedure for determining extreme pressure properties of solid bonded films under high-frequency linear-oscillation motion using the SRV test machine. This laboratory test method can be used to quickly determine extreme pressure properties of parts coated with solid bonded film at selected temperatures specified for use in unlubricated applications where high-speed vibrational or start-stop motions are present with high Hertzian point contact. This test method has found wide application in qualifying solid bonded films used in automotive door lock mechanisms, hinge joints, bolts, and in aerospace. This test method is a material and application oriented approach for characterizing the tribological behavior using random, discrete, and constant parameter combinations. Users of this method should determine whether results correlate with field performance or other applications.

TEST SUMMARY

This test method is performed on an SRV test machine using a steel test ball oscillating against a steel test disk with lubricant between them. The test load is increased in 100-N increments until seizure occurs. The load, immediately prior to the load at which seizure occurs, is measured and reported.

Test frequency, stroke length, temperature, and ball and disk material can be varied to simulate field conditions. The test ball yields point-contact geometry. To obtain line or area contact, test pieces of differing configurations can be substituted for the test balls.

TEST PRECISION

Test Time, min	1	3	6
Repeatability	0.25 *X*	0.44 *X*	0.45 *X*
Reproducibility	0.85 *X*	0.48 *X*	0.29 *X*

Where *X* is the average of two results in N.

This method has no bias because the load-carrying capacity can be defined only in terms of this test method.

EXTRACTABLES SOLVENT EXTRACTABLES IN PETROLEUM WAXES:
D 3235

EXPLANATION

The solvent extractables in a wax may have significant effects on several of its properties such as strength, hardness, flexibility, scuff resistance, coefficient of friction, coefficient of expansion, melting point, and staining characteristics. Whether these effects are desirable or not depends on the intended use of the wax.

TEST SUMMARY

The sample is dissolved in a mixture of 50 volume % methyl ethyl ketone and 50 volume % toluene. The solution is cooled to −32°C(−25°F) to precipitate the wax, then filtered. The solvent extractables content is determined by evaporating the solvent from the filtrate and weighing the residue.

TEST PRECISION

In the extractables range 15 to 55 %, the test method has a repeatability of 2 % and a reproducibility of 5 %.

This test method has no bias.

FILTER PLUGGING TENDENCY OF DISTILLATE FUEL OILS:
D 2068

EXPLANATION

Causes of poor filterability might include fuel degradation products, contaminants picked up during storage or transfer, or interaction of the fuel with the filter media. A change in filter performance after storage or pretreatment can be indicative of changes in fuel condition. This test method is intended for use in evaluating distillate fuel cleanliness in those applications that demand a high throughput per installed filter. This test method is applicable to fuels within the viscosity range of 1.50 to 6.00 cSt at 40°C. This test method is not applicable to fuels that are not clear and bright because water interferes with the measurement of filter plugging. Relative tendency of fuels to plug filters may vary depending on filter porosity and structure, and may not always correlate with the results from this test method.

TEST SUMMARY

A sample of fuel is passed at a constant flow rate (20 mL/min) through a glass fiber filter medium. The pressure drop across the filter is monitored during the passage of a fixed volume of test fuel. If a prescribed maximum pressure drop is reached before the total volume of fuel is filtered, the actual volume of fuel filtered at the time of maximum pressure drop is recorded. The apparatus is required to be calibrated at intervals.

TEST PRECISION

This test method has a repeatability of 0.27 (FPT − 1) and a reproducibility of 1.405 (FPT − 1) where FPT is the normalized result.

This test method has no bias.

FILTERABILITY OF AVIATION TURBINE FUEL:
D 6824

EXPLANATION

This test method is intended for use in the laboratory or field in evaluating cleanliness of aviation turbine fuel. A change in filtration performance after storage, pretreatment, or commingling can be indicative of changes in fuel condition. Relative filterability of fuels may vary, depending on filter

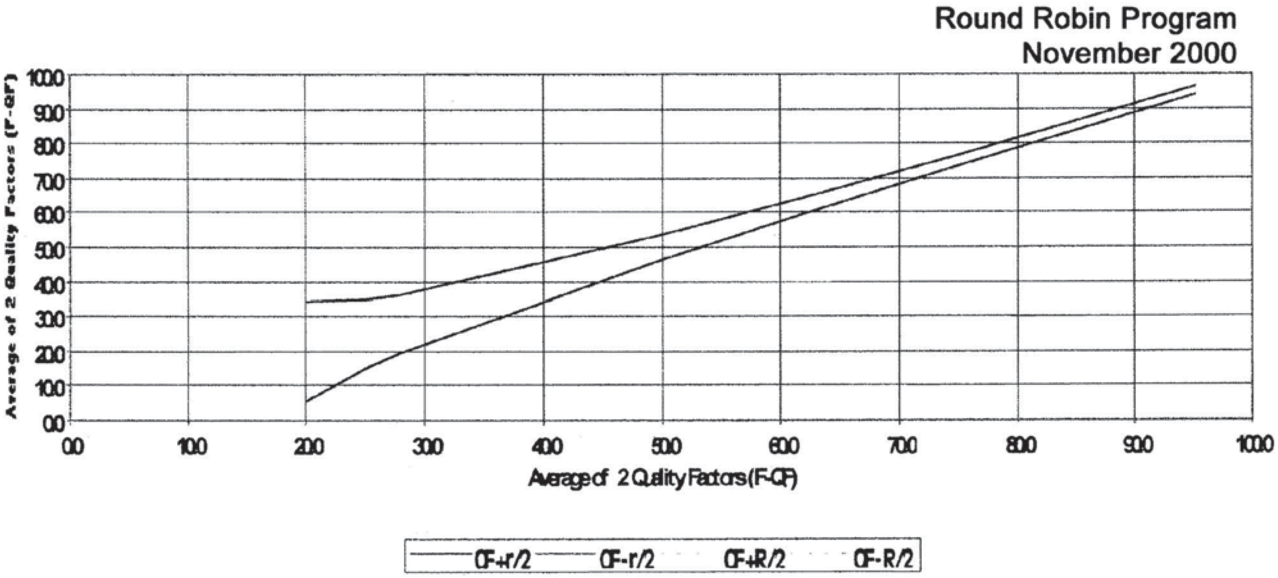


FIG. 8—*Precision for Emcee Model-1143 Jet Fuel @ .65 micron Filter Pore Size (Repeatability (r) & Reproducibility (R)).*

porosity and structure, and may not always correlate with results from this test method. Causes of poor filterability in industrial refinery filters include fuel degradation products, contaminants picked up during storage or transfer, incompatibility of commingled fuels, or interaction of the fuel with the filter media. Any of these could correlate with orifice or filter system plugging, or both. ASTM specification fuels falling within the scope of this method are Specifications D 1655 and D 6615, and the military fuels covered in the military specifications. This test method is not applicable to fuels that contain undissolved water.

TEST SUMMARY

A sample is passed at a constant rate (20 mL/min) through a standard porosity filter medium. The pressure drop across the filter and the volume of filtrate are monitored. The test is concluded either when the pressure drop across the filter exceeds 104 kPa, or when 300 mL have passed through the filter.

TEST PRECISION

This test method has no bias because the filterability quality factor is defined only in terms of this test method.

FILTERABILITY OF DIESEL FUELS BY LTFT: D 4539

EXPLANATION

The low temperature flow test (LTFT) results are indicative of the low temperature flow performance of fuel in some diesel vehicles. This test method is especially useful for the evaluation of fuels containing flow improver additives.

TEST SUMMARY

The temperature of a series of test specimens of fuel is lowered at a prescribed cooling rate. At the commencing temperature and at each 1°C interval thereafter, a separate specimen from the series is filtered through a 17-μm screen until a minimum LTFT pass temperature is obtained. The minimum LTFT pass temperature is the lowest temperature, expressed as a multiple of 1°C, at which a test specimen can be filtered in 60 s or less.

TEST PRECISION

Repeatability:	2°C
Reproducibility:	4°C

No statement of bias can be made for this test method.

FILTERABILITY OF DISTILLATE FUEL OILS: D 6426

EXPLANATION

A change in filtration performance after storage, pretreatment, or commingling can be indicative of changes in fuel condition. Relative filterability of fuels may vary depending on filter porosity and structure and may not always correlate with results from this test method. Causes of poor filterability in industrial/refinery filters include fuel degradation products, contaminants picked up during storage or transfer, incompatibility of commingled fuels, or interaction of the fuel with the filter media. Any of these could correlate with orifice or filter system plugging, or both.

This test method is applicable to distillate fuel oils within the viscosity range from 1.70 to 6.20 cSt at 40°C. ASTM specification fuels falling within the scope of this test method are Specification D 396, Grade Nos. 1 and 2, Specification D 975, Grades 1-D, low sulfur 1-D, 2-D, and low sulfur 2-D, and Specification D 2880, Grade Nos. 1-GT and 2-GT. This test method is not applicable to fuels that contain undissolved water.

TEST SUMMARY

A sample is passed at a constant rate (20 mL/min) through a standard porosity filter medium. The pressure drop across the filter and the volume of filtrate are monitored. The test is concluded either when the pressure drop across the filter exceeds 170 kPa or when 300 mL have passed through the filter. It is not necessary to verify apparatus performance prior to each test. But verification of the apparatus is required when there is a doubt of a test result, or when the apparatus has not been used for three months or more.

TEST PRECISION

Precision for Diesel, Home Heating, and Blended Bio-diesel Fuels (5.0 μ Filter).

Maximum Allowable Difference Between Two F-QF Values		
Two F-QF Values, avg	Repeatability	Reproducibility
10	10	12
20	11	14
30	12	15
40	13	16
50	14	18
60	15	19
70	16	21
80	17	22
90	19	24
100	20	25

FILTERABILITY OF ENGINE OILS AFTER TREATMENT WITH WATER:
D 6794

EXPLANATION

Usually some of the combustion products of an internal combustion engine penetrate into the engine lubricant and are retained in it. When the engine is stored over a long period of time after running for a short period of time, the by-products of combustion may be retained in the oil in a liquefied state. Under these circumstances precipitates can form that impair the filterability of the oil next time the engine is run. This test method subjects the test oil and the new oil to the same treatments such that the loss of filterability can be determined. The test method requires that a reference oil also be tested and results reported. Two oils for this purpose are available from ASTM Test Monitoring Center. One oil gives a high data value, and the other gives a low value for this test.

With the use of ASTM TMC reference oils, a laboratory can assess whether their use of this test method gives statistically equivalent results to those obtained by other laboratories. Also, various agencies require that a laboratory utilize the TMC services in seeking qualification of oils against specifications.

TEST METHOD SUMMARY

The test oil is treated with deionized water and is heated to 70°C for 6 h, followed by storage at room temperature. The sample is filtered and the flow rate is calculated determining the engine oil filterability characteristics.

TEST PRECISION

Repeatability:	11.1 %
Reproducibility:	25.4 %

These precision estimates should be used for judging the acceptability of 25 μm filter results. Since there is no accepted reference material suitable for determining the bias for this test method, bias cannot be determined.

FILTERABILITY OF ENGINE OILS AFTER TREATMENT WITH WATER AND DRY ICE:
D 6795

EXPLANATION

See D 6794 regarding the significance of this test method.

TEST SUMMARY

The test oil is treated with deionized water and dry ice, and is heated for 30 min at 70°C, followed by storage at room temperature. The sample is filtered and the flow rate is calculated determining the engine oil filterability characteristics.

TEST PRECISION

Repeatability:	11.4 %
Reproducibility:	23.6 %

This precision estimate is based on use of 15 μm filter results. Since there is no accepted reference materials suitable for determining the bias of this test method, bias cannot be determined.

DISCRIMINATION BETWEEN FLAMMABILITY RATINGS: D 6668

EXPLANATION

This test method covers a means for the discrimination between solid and liquid materials which will not burn in air when exposed to a temperature of 1500°F for a period of 5 min and those which will ignite and burn under the same conditions. This test method can be applied to any substance which is solid or liquid at ambient temperature and pressure. This test method provides a means for the classification of flammability hazard of material of hazard degrees (F=0) and (F=1) or both, in accordance with the definitions of degrees of flammability hazard as defined by NFPA 704 para, 3.2.1.

This test method should be used subject to the limitations that no single fire hazard property such as flash point, ignition temperature, or the performance under the conditions of this test method shall be used to describe or appraise the fire hazard or fire risk of a material, product, assembly or system under actual fire conditions. Fire hazard properties measured under controlled laboratory conditions may, nevertheless, be employed to describe properly the response of materials, products, assemblies or systems under said controlled conditions. Properties measured under controlled laboratory conditions may be used as elements of hazard or risk assessment only when such assessment takes into account all of the factors that are pertinent to the evolution of the fire hazard of a given situation.

TEST SUMMARY

Small, measured amounts of the sample are placed on a stainless steel surface heated to 1500°F. Reactions which occur during the 5 min interval, thereafter, are observed and recorded. A material which does not exhibit any evidence of burning as defined herein under the conditions of the test procedure may be classified as Flammability Degree of Hazard Zero material (F=0) provided other properties of the material are not such as to require a higher degree of classification.

TEST PRECISION

No information is available about either precision or bias of this test method since the results are nonquantitaive.

FUEL INJECTOR SHEAR STABILITY TEST (FISST) FOR POLYMER CONTAINING FLUIDS:
D 5275
(Equivalent Test Methods: IP 294 and DIN 51382)

EXPLANATION

This test method evaluates the percent viscosity loss for polymer-containing fluids resulting from polymer degradation in the high shear nozzle device. Minimum interference from thermal or oxidative effects are anticipated. This test method is not intended to predict viscosity loss in field service for different polymer classes or for different field equipment. Some correlation for a specific polymer type in specific field equipment can be possible. Test Method D 2603 has been used for similar evaluation of this property. No detailed attempt has been undertaken to correlate the results by sonic and the diesel injector methods.

TEST SUMMARY

The polymer containing fluid is passed through a diesel injector nozzle at a shear rate that causes the less shear stable polymer molecules to degrade. This reduces the kinematic viscosity of the fluid under test. The reduction in kinematic viscosity, reported as a percent loss of the initial kinematic viscosity, is a measure of the shear stability of the polymer containing fluid.

TEST PRECISION

Repeatability:	1.19 %
Reproducibility:	5.22 %

No estimate of bias can be made for this test method.

FLASH POINT:

GENERAL

The lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of a test flame causes the vapor of a sample to ignite under specified test conditions. The sample is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the sample. Occasional appearance of a blue halo or an enlarged flame, close to the actual flash point is not considered a true flash point.

Flash point measures the tendency of a sample to form a flammable mixture with air under controlled laboratory conditions. Flash point data are used in shipping and safety regulations to define “flammable” and “combustible” materials. Flash point data can also indicate the possible presence of highly volatile and flammable material in a relatively nonvolatile or nonflammable material.

Erroneously high flash points can be obtained when precautions are not taken to avoid the loss of volatile material. Samples should not be stored in plastic bottles, since the volatile material may diffuse through the walls of the container. The containers should not be opened unnecessarily. The samples should not be transferred between containers unless the sample temperature is at least 20°F (11°C) below the expected flash point.

Flash point should not be confused with autoignition temperature (ASTM Test Method E 659), which measures spontaneous combustion with no external source of ignition.

There are five different methods of determining flash point (see Table 5):

	ASTM Test Methods
Cleveland Open Cup	D 92
Pensky Martens Closed Cup Tester	D 93
Tag Closed Tester	D 56
Small Scale Closed Tester	D 3828
Continuously Closed Cup Flash Point	D 6450

TABLE 9—*Comparison of Flash Point Test Methods.*

Test Method	D 56	D 92	D 93	D 3828	D 6450	D 7094	D 7236
Technique	Tag Closed Tester	Cleveland Open Cup	Pensky Martens Closed Cup Tester	Small Scale Closed Tester	CCFP	MCCFP	SSCCT
Scope	Liquids of viscosities <5.5 cSt at 40°C, or <9.5 cSt at 25°C; flash points <93°C	All petroleum products with flash points in 79–400°C range	Fuel oils, lube oils, solid suspension liquids with viscosity >5.5 cSt 40°C. Range at 40 to 360°C		Fuel oils, lube oils Solvents, and other Petroleum liquids	Fuel oils, lube oils, Solvents, and other Petroleum liquids	Aviation turbine fuel, diesel fuel, kerosene in temperature range 40–135°C
Limitations	Not applicable to cut-back asphalts and liquids beyond scope	Not applicable for fuel oils	Use Procedure B for viscous liquids				
Sample size	50 mL	75 mL	75 mL	2–4 mL	1 mL	2 mL	2 mL 0.01285 $(x+40)$
Repeatability	<60°C:1.2°C	8°C	A: $0.035X$°C	20–70°C:0.5°C	1.9°C	4.1°C	
	>60°C:1.6°C		B: 5°C	>70°C:$0.022M^{0.9}$°C			
Reproducibiity	<60°C:4.3°C	18°C	A: $0.078X$°C	20–70°C: $0.03(M)+29)$°C	3.1°C	5.5°C	$0.02482(x+40)$
	>60°C:5.8°C		B: 10°C	>70°C:$0.083M^{0.0}$°C			
Bias	None	None	Not Determined	None	N/A	N/A	N/A

FLASH POINT BY CONTINUOUSLY CLOSED CUP TESTER (CCFP):

D 6450

EXPLANATION

This test method covers the determination of the flash point of fuel oils, lube oils, solvents, and other liquids by a continuously closed cup tester. The measurement is made on a test specimen of 1 mL. This test method utilizes a closed but unsealed cup with air injected into the test chamber. This test method is suitable for testing samples with a flash point from 10 to 250°C. Flash points below 10°C and above 250°C can be performed; however, the precision has not been determined at these temperatures.

TEST SUMMARY

The lid of the test chamber is regulated to a temperature of at least 18°C below the expected flash point. A 1-mL test specimen is introduced into the sample cup, ensuring that both specimen and cup are at a temperature of at least 18°C below expected flash point, cooling if necessary. The cup is then raised and pressed onto the lid of specified dimensions to form the continuously closed but unsealed test chamber with an overall volume of 4 mL. The lid is heated at a prescribed, constant rate. An arc of defined energy is discharged inside the test chamber at regular intervals. After each ignition, 1.5 mL of air is introduced into the test chamber to provide the necessary oxygen for the next flash test. After each arc, the instantaneous pressure increase above the ambient barometric pressure inside the test chamber is monitored. When the pressure increase exceeds a defined threshold, the temperature at that point is recorded as the uncorrected flash point.

TEST PRECISION

Repeatability:	1.9°C
Reproducibility:	3.1°C

Bias has not been determined.

FLASH POINT BY CLEVELAND OPEN CUP:

D 92

(Equivalent Test Methods: IP 36, ISO 2592, DIN 51376, JIS K 2265, and AFNOR T60-118)

EXPLANATION

This test method covers the determination of the flash and fire point of all petroleum products except fuel oils and those having an open cup flash <175°F(79°C).

TEST SUMMARY

The test cup is filled to a specified level with sample. The temperature is rapidly increased at first, and then at a slow constant rate as the flash point is approached. At specified intervals a small test flame is passed across the cup. The lowest temperature at which the vapors above the surface of the liquid ignites is taken as the flash point. The test is continued until the application of the test flame causes the oil to ignite and burn for at least 5 s. This temperature is called the fire point.

TEST PRECISION

	Repeatability	Reproducibility
Flash point, °F (°C)	15(8)	32(18)
Fire point, °F (°C)	15(8)	25(14)

FLASH POINT BY MCCFP TESTER: D 7094

EXPLANATION

The flash point measurement is one measure of the tendency of the test specimen to form a flammable mixture with air under controlled laboratory conditions. Flash point is used in shipping and safety regulations to define flammable and combustible materials and for classification purposes. This test method can be used to measure and describe the properties of materials in response to heat and an ignition source under controlled laboratory conditions and shall not be used to describe or appraise the fire hazard or fire risk of materials under actual fire conditions. Flash point can also indicate the possible presence of highly volatile and flammable materials in a relatively nonvolatile or nonflammable materials, such as the contamination of lubricating oils by small amounts of diesel fuel or gasoline. This test method was designed to be more sensitive to potential contamination than Test Method D 6450.

This test method covers the determination of flash point of fuels, lube oils, solvents, and other liquids by a continuously closed cup tester utilizing a specimen size of 2 mL, a cup size of 7 mL, with a heating rate of 2.5°C per minute. Test Method D 6450 uses a 1 mL sample, a cup size of 4 mL, and a heating rate of 5.5°C per minute.

No statistically significant bias was observed between this test method and Test Method D 93 PMCC flash point method in the temperature range from 35 to 150°C.

This test method is a dynamic method and depends on definite rates of temperature increase. This test method utilizes a closed but unsealed cup with air injected into the test chamber. This test method is suitable for testing samples with a flash point from 35 to 225°C. If the user's specification requires a flash point method other than this method, neither this method or any other test method should be substituted for the prescribed test method without obtaining comparative data and an agreement from the specifier. This fact is valid for all flash point test methods.

TEST SUMMARY

The lid of the test chamber is regulated to a test temperature at least 18°C below the expected flash point. A 2 ± 0.2 mL test specimen of a sample is introduced into the sample cup. The cup is then raised and pressed onto the lid of specified dimensions to form the continuously closed but unsealed test chamber with an overall volume of 7.0 ± 0.3 mL. After closing the test chamber, the temperatures of the test specimen and the regulated lid are allowed to equilibrate to within 1°C. Then the lid is heated at a prescribed, constant rate. A high voltage arc of defined energy is discharged inside the test chamber at regular intervals. After each ignition, a variable amount of air is introduced into the test chamber to provide the necessary oxygen for the next flash test. The pressure inside the continuously closed but unsealed test chamber remains at ambient barometric pressure except for the short time during the air introduction and at a flash point. After each arc application, the instantaneous pressure increase above the ambient barometric pressure inside the test chamber is monitored. When the pressure increase exceeds 20 kPa, the temperature at that point is recorded as the uncorrected flash point, which is then corrected to barometric pressure.

TEST PRECISION

Repeatability:	4.1°C
Reproducibility:	5.5°C

These precision statements are valid in the flash point range between 35 and 225 °C.

Since there is accepted reference materials suitable for determining bias for this test procedure, bias has not been determined.

FLASH POINT BY SMALL SCALE CLOSED CUP TESTER (RAMP METHOD): D 7236

EXPLANATION

This test method covers the determination of the flash point of aviation turbine fuel, diesel fuel, kerosene, and related products in the temperature range of 40 to 135°C by a small scale closed cup apparatus. This test method is not applicable to liquids contaminated by traces of highly volatile materials.

This test method is a dynamic method and depends on a definite rate of temperature increase. If the product specification requires a specific flash point method, neither this or any other method should be substituted without agreement between the buyer and the seller of the product.

TEST SUMMARY

A 2 ± 0.1 mL test specimen is introduced into the test cup that is then heated automatically at a constant rate of 2 ± 0.5°C/min. A gas test flame is directed through an opening shutter, in the test cup lid, at specified temperature intervals until a flash is detected by the automatic flash detector.

TEST PRECISION

Repeatability:	0.01285(x + 40)
Reproducibility:	0.02482(x + 40)

Where x is the average of results being compared in the flash point range of 40 to 135°C.

Since there is no accepted reference material for this test method, bias of the method cannot be determined.

FLASH POINT BY PENSKY-MARTENS CLOSED TESTER: D 93

(Equivalent Test Methods: IP 34, ISO 2719, DIN 51758, JIS K 2265, and AFNOR M07-019)

EXPLANATION

This method is used for the analysis of fuel oils, lube oils, suspension of solids, liquids that tend to form a surface film under test conditions, and other liquids.

TEST SUMMARY

The sample is heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which the vapor above the sample ignites.

There are two procedures allowed which differ in their heating rate and the stirrer speed of the sample.

	Procedure A	Procedure
Samples	Ordinary liquids	Suspension of solids and highly viscous materials
Heating Rate	5°C-6°C/min	1°C-1.5°C/min
Stirrer Speed	90-120 r/min	250 ± 10 r/min

TEST PRECISION

	Repeatability	Reproducibility
Procedure A	0.029 X°C	0.071 X°C
Procedure B		
Residual Fuel Oil	2°C	6°C
Other Types	5°C	10°C

Where X is the result in °C

FLASH POINT SMALL SCALE CLOSED TESTER: D 3828

(Equivalent Test Methods: IP 303 and ISO 3679)

EXPLANATION

These test methods cover procedures for the determination of the flash point by a small scale closed tester. The procedures may be used to determine the actual flash point temperature of a sample or whether a product will or will not flash at a specified temperature (flash/no flash).

TEST SUMMARY

Method A: Flash/No Flash Test—A portion of a sample is introduced by a syringe into the cup of the selected apparatus that is set and maintained at the specified temperature. After a specific time a test flame is applied and an observation made as to whether or not a flash occurred.

Method B: Finite (or Actual) Flash Point—A portion of a sample is introduced into the cup of the selected apparatus that is maintained at the expected flash point. After a specified time, a test flame is applied and an observation made as to whether or not a flash occurred.

The portion is removed from the cup, the cup cleaned, and the cup temperature adjusted 9°F (5°C) lower or higher depending on whether or not a flash occurred previously. A fresh portion is introduced and tested. This procedure is repeated until the flash point is established within 9°F (5°C).

The procedure is then repeated at 2°F (1°C) intervals until the flash point is determined to the nearest 2°F (1°C).

If improved accuracy is desired, the procedure is repeated at 1°F (0.5°C) intervals until the flash point is determined to the nearest 1°F (0.5°C).

TEST PRECISION

Range	Repeatability	Reproducibility
68 to 158°F(20 to 70°C)	0.9°F(0.5°C)	$0.03+22$°F $[0.03(M+29)$°C$]$
Above 158°F(70°C)	$0.0117\,M$°F ($0.022\,M^{0.9}$°C)	$0.045\,M$°F ($0.083\,M^{0.9}$°C)

Where M is the mean of the two results.

FLASH POINT TAG CLOSED TESTER:

D 56

(Equivalent Test Methods: DIN 51411, JIS K 2580, and AFNOR M07-003)

EXPLANATION

This test method covers the determination of the flash point, by Tag closed tester, of liquids with a viscosity of <5.5 cSt at 104°F(40°C), or <9.5 cSt at 77°F(25°C) and a flash point <200°F(93°C), except cutback asphalts that tend to form a surface film, and materials which contain suspended solids. For other liquids Test Method D 93 should be used.

TEST SUMMARY

The sample is placed in the cup of the tester and with the lid closed, heated at a slow constant rate. A small flame of specified size is directed into the cup at regular intervals. The flash point is taken as the lowest temperature at which the application of the test flame causes the vapor above the sample to ignite.

TEST PRECISION

Flash Point, °F (°C)	Repeatability	Reproducibility
<60°C(140°F)	1.2°C(2.0°F)	4.3°C(9°F)
=/>60°C(138.2°F)	1.6° (3.0°F)	5.8°C(10°F)

MAXIMUM FLOCCULATION RATIO AND PEPTIZING POWER IN RESIDUAL AND HEAVY FUEL OILS:

D 7060

EXPLANATION

Asphaltenes are naturally occurring materials in crude petroleum and petroleum products containing residual material. The asphaltenes are usually present in colloidal suspensions, but they may agglomerate and flocculate if the suspension of asphaltene molecules is disturbed through excess stress or incompatibility. This test method provides compatibility parameters, which can be used to assess stability reserve and compatibility. This method covers a procedure for quantifying the maximum flocculation ratio of the asphaltenes in the oil and the peptizing power of the oil medium, by an automatic instrument using an optical device. This test method is applicable to atmospheric or vacuum distillation residues, thermally cracked residue, intermediate and finished residual fuel oils, containing at least 1 mass % asphaltenes. This test method has not been developed for asphalts. This test method is applicable to products typical of Specification D 396—Grades 5L, 5H, and 6, and Specification D 2880—Grades 3-GT and 4-GT.

A blend is considered stable when the blend's peptizing power is higher than the blend's maximum flocculation ratio. Both of them can be calculated using empirical blend rules.

Refineries and terminal owners can prevent the flocculation of asphaltenes due to incompatibility by assessing the compatibility of fuels beforehand.

TEST SUMMARY

Six portions of the sample are diluted in various ratios with 1-methylnaphthalene. Each solution is inserted into the automatic apparatus, and titrated with cetane until flocculation of asphaltenes is detected by optical probe. The first two solutions are titrated with cetane in coarse determinations in which the flocculation ratio is decreased in 5 % steps. The coarse determinations help to establish suitable starting values for the fine determinations, in which the flocculation ratio is decreased in 1 % steps. The flocculation ratios at critical dilution, measured during the fine determinations, are used to calculate the maximum flocculation ratio of the sample's asphaltenes and the peptizing power of the sample's oil medium.

An optical probe detects the formation of flocculated asphaltenes. The start of flocculation is interpreted when a significant and sustained increase in rate-of-change of signal, as measured by the optical probe, ensures flocculation is in progress. The start of flocculation can be detected unambiguously when the sample contains at least 1 % mass asphaltenes as measured by Test Method D 6560.

High content of insoluble inorganic matter (sediment) has some interference in this test method. In this case, the insoluble matter shall be removed by filtration according to Test Method D 4870. The presence of wax, present in the paraffinic crudes or fuels from such crudes, does not interfere.

TEST PRECISION

In the flocculation ratio range from 16 to 57 and the peptizing power range from 27 to 96, the repeatability was found to be:

Maximum Flocculation Ratio	3
Peptizing Power	6

The reproducibility of this test method has not yet been determined.

This test method has no bias because the results of this test are defined only in terms of this test method.

FOAMING TENDENCY IN AQUEOUS MEDIA
D 3519 AND D 3601

The results from these tests are useful as guides in determining the tendency of a water-based metal working coolant to produce foam under low or high shear conditions. No correlation with changes in heat transfer, pumpability, or other factors affected by foam is intended. The foam generated by any given industrial process depends on the method by which the foam is generated, and may not be directly proportional to that produced by this controlled laboratory test method. The foam generated at the specified test temperature will not necessarily predict the foaming tendency of the liquid at some other use temperature. There are two test methods for this analysis. Both measure the increase in volume of a low viscosity aqueous liquid (<3 cSt at 40°C) due to its tendency to foam under high shear conditions in Test Method D 3519 and at low shear conditions in Test Method D 3601.

TEST SUMMARY

In Test Method D 3519 procedure, the test fluid is blended for 30 s using a commercial type blender at 25 ± 1°C agitating between 4000 and 13 000 r/min. The preferred range would be 8000 ± 1000 r/min.

In Test Method D 3601 procedure, the test fluid is vigorously shaken at 25 ± 1°C.

At the end in both the procedures the increase in height of the test fluid is measured.

TEST PRECISION

Test Method	Measurement	Repeatability	Reproducibility
D 3519	Maximum Foam	25 Mean %	70 Mean %
	Residual Foam	24	43
D 3601	Maximum Foam	7.0 mm	72 % relative
	Residual Foam	Not Known	Not Known

The bias of either of these two test methods is not available.

FOAMING TENDENCY OF LUBRICATING OILS: **D 892**

(Equivalent Test Methods: IP 146, ISO 6247, DIN 51566, and JIS K 2518)

EXPLANATION

The tendency of oils to foam can be a serious problem in systems such as high-speed gearing, high-volume pumping, and splash lubrication. Inadequate lubrication, cavitation, and overflow loss of lubricant may lead to mechanical failure. This test method is valuable in the selection of oils suitable for such operating conditions.

Norton Stones and Mott metal diffusers may be used. Both give equivalent results for most oil samples; though there may be some exceptions. Recent industry experience shows that even the so-called "calibrated" Norton Stones fail to pass porosity and permeability requirements sometimes 100 % of the time. On the other hand, Mott metal diffusers have a 100 % acceptance rate. Hence, it is preferable to use the metal diffusers, even though they cost more than the stones.

ASTM has recently formed an adhoc committee to recommend on the choice of diffusers. It is expected that this group will recommend that the stone diffusers should not be allowed in this test.

TEST SUMMARY

The sample maintained at a temperature of 24°C(75°F) is blown with air at a constant rate for 5 min, then allowed to settle for 10 min. The volume of foam is measured at the end of both periods. The test is repeated on a second sample at 93.5°C(200°F), and then, after collapsing the foam, at 24°C (75°F).

It is critical that the diffusers be first tested for maximum pore diameters and permeability as given in the mandatory annex to the test method. A schedule for cleaning and replacing the diffusers should be established. A 1994 ASTM cross-check showed serious disagreement in testing the diffuser porosity.

TEST PRECISION

See Figs. 9 and 10 .

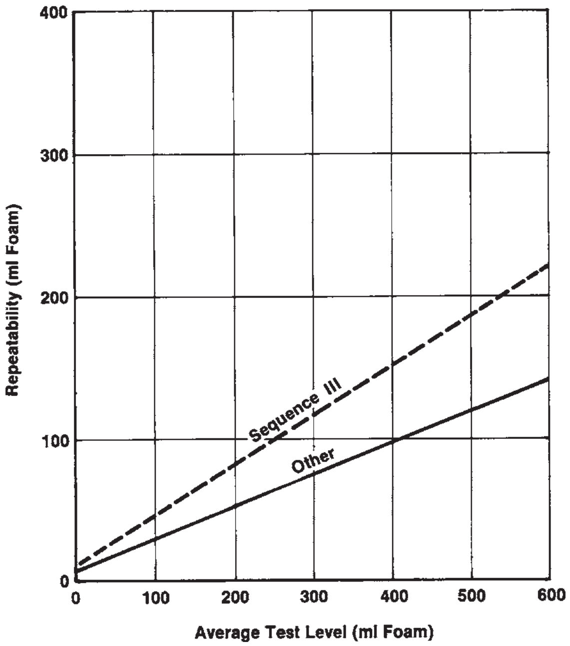


FIG. 9—*Precision Chart—Repeatability.*

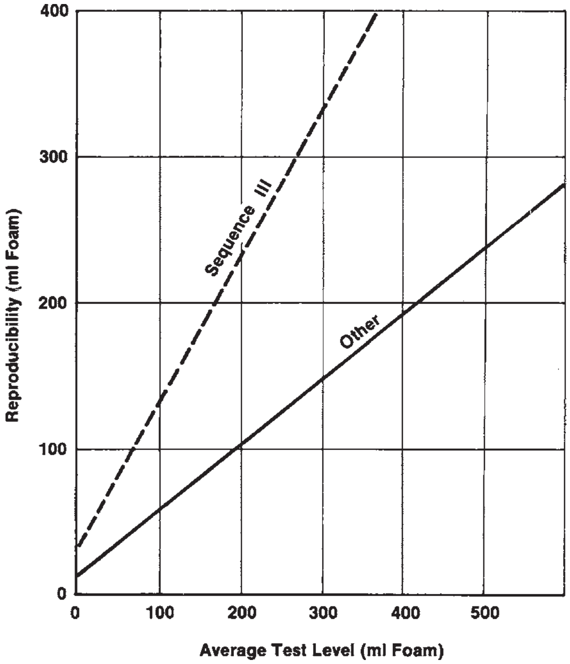


FIG. 10—*Precision Chart—Reproducibility.*

FOAMING TENDENCY HIGH TEMPERATURE FOAMING TENDENCY:
D 6082

EXPLANATION

This test commonly known as the “Sequence IV” foam test is the high temperature version of Test Method D 892. This was originally a Ford test, which has now been accepted by all OEMs in a slightly modified form. The test determines the foaming characteristics of lubricating oils at 150°C. This is expected to mimic operations in high-speed gearing, high volume pumping, and splash lubrication.

TEST SUMMARY

A measured quantity of sample is heated to 49°C for 30 min, allowed to cool to room temperature, then tested for foaming characteristics at 150°C. The sample is transferred to a 1000-mL graduate cylinder and aerated with a metal (Mott) diffuser at 200 mL/min dry air for 5 min. The amount of foam generated prior to disconnecting the air, the amount of foam at 5 s after disconnecting the air, the time for the foam to collapse, and the amount of foam remaining after 10 min is determined.

TEST PRECISION[1]

[1] This table is Table 1 in Test Method D 6082-06.

	Repeatability	Reproducibility	Range
Foaming Tendency (5 min)	$3.0X^{.5}$	$8.2X^{.5}$	8.1–440 mL
Total Volume Increase	$0.82X^{.8}$	$1.8X^{.8}$	17–270 mL
Total Foam	$2.3X^{.5}$	$10.X^{.5}$	99–530 mL
Static Foam	$1.4X^{.6}$	$5.8X^{.6}$	48–510 mL
Kinetic Foam	$0.60\ (X-150)$	$0.63\ (X+150)$	12–160 mL
Foam Stability			
5 s (after air disconnect)	$2.3X^{.5}$	$10.X^{.5}$	11–480 mL
15 s (after air disconnect)	$2.3X^{.5}$	$10.X^{.5}$	0–440 mL
1 min (after air disconnect)	$2.3X^{.5}$	$10.X^{.5}$	0–300 mL
10 min (after air disconnect)			0 mL
Collapse Time	$0.34X$	$0.83X$	9–460 s

FREEZING POINT OF AVIATION FUELS:
D 2386
(Equivalent Test Methods: IP 16, ISO 3013, DIN 51421, JIS K 2276, and AFNOR M07-048)

GENERAL

The freezing of an aviation fuel is an index of the lowest temperature of its utility for certain applications. Solid hydrocarbon crystals can restrict the flow of fuel in the fuel system of the aircraft. The temperature of the fuel in the aircraft tank normally decreases during flight depending on aircraft speed, altitude, and flight duration. The freezing point of the fuel must always be lower than the minimum operational fuel temperature. Petroleum blending operations require precise freezing point measurements. The freezing point is a requirement of specifications D 910 and D 1655. Typical freezing point of aviation fuels is in the range −40 to −65°C.

There are three test methods available for this analysis. All three have been found to give equivalent results. However, when a specification calls for a specific test, only that test must be used.

A measured fuel sample is placed in a jacketed sample tube, also holding a thermometer and a stirrer. The tube is placed in a vacuum flask containing the cooling medium. Various coolants used are acetone, methyl-, ethyl- or iso-propyl alcohols, solid carbon dioxide, or liquid nitrogen. As the sample cools, it is continuously stirred. The temperature at which the hydrocarbon crystals appear is recorded. The jacketed sample is removed from the coolant and allowed to warm, stirring it continuously. The temperature at which the crystals completely disappear is recorded.

TEST PRECISION

Repeatability:	0.8°C
Reproducibility:	2.3°C

FREEZING POINT OF AVIATION FUELS (AUTOMATIC FIBER OPTICAL METHOD):
D 7154
(Equivalent Test Method: IP 528)

EXPLANATION

The freezing point of an aviation fuel is an index of the lowest temperature of its utility for certain applications. Solid hydrocarbon crystals can restrict the flow of fuel in the fuel system of the aircraft. The temperature of the fuel in the aircraft tank normally decreases during flight depending on aircraft speed, altitude, and flight duration. The freezing point of the fuel must always be lower than the minimum operational fuel temperature. Typical aviation fuel has freezing point temperatures in the −60 to −40°C range. Petroleum blending operations require precise measurement of the freezing point. The measuring range of the apparatus used in this test method is from −70 to 0°C. This test method expresses results with a resolution of 0.1°C. This test method eliminates most of the operator time and judgment required by Test method D 2386. However, when the specification requires the use of Test Method D 2386, do not substitute this test method or any other method.

TEST SUMMARY

A 25 mL portion of the test specimen is inserted into a test chamber, after which it is cooled while being continuously stirred and monitored by a fiber optical system. The temperature of the specimen is measured with an electronic temperature measuring device. When crystal formation is detected in the specimen, the temperature is recorded and the specimen in the test chamber is warmed, while being continuously stirred and monitored by the optical system, until the crystals in the specimen completely disappear. The temperature of the specimen when the last crystals disappear is recorded as the freezing point of the specimen.

TEST PRECISION

Repeatability:	0.5°C
Reproducibility:	1.9°C

Because there are no liquid hydrocarbon mixtures of known freezing point, which simulate aviation fuels, bias cannot be established. No relative bias between Test Method D 2386 and this test method was observed.

FREEZING POINT OF AVIATION FUELS (AUTOMATIC LASER METHOD):
D 7153
(Equivalent Test Method: IP 5290)

EXPLANATION

See explanation in Test Method D 7154.

This test method covers the temperature range −80 to 20°C.

TEST SUMMARY

A specimen is cooled at a rate of 10±0.5°C/min while continuously being illuminated by a laser light source. The specimen is continuously monitored by optical crystal and opacity detectors for the first formation of solid hydrocarbon crystals. Once the hydrocarbon crystals are detected by both sets of optical detectors, the specimen is then warmed at a rate of 3 ± 0.5 °C/min. When initial opacity in the specimen disappears, the specimen is then warmed at a rate of 12 ± 1 °C/min. The specimen temperature at which the last hydrocarbon crystals return to the liquid phase, as detected by the crystal detector, is recorded as the freezing point.

In certain circumstances, as measured by the apparatus, the specimen is reheated to approximately 10 °C, then cooled at the rate mentioned above until the hydrocarbon crystals are detected by the crystal detector. The specimen is then warmed at a rate of 12 ± 1 °C/min, until the last hydrocarbon crystals return to the liquid phase. The specimen at which the last hydrocarbon crystals return to the liquid phase, as detected by the crystal detector, is recorded as the freezing point.

TEST PRECISION

Repeatability:	0.6 °C
Reproducibility:	0.9 °C

Because there are no liquid hydrocarbons mixtures of known freezing point, which simulate aviation fuels, bias cannot be established. A systematic bias was observed between this method and Test Methods D 2386 or IP 16.

Mean of the result by D 2386 or IP 16 = X − 0.347 where X is the mean of result by D 7153. However, the relative bias is within the reproducibility of both test methods.

FREEZING POINT:
D 5901
(Equivalent Test Method: IP 434)

This is an automated optical method used for the temperature range 0 to −70 °C. Results by this test method have a resolution of 0.1 °C. A 25-mL portion of the fuel is placed in a test chamber, which is cooled while continuously being stirred and monitored by an optical system.

The temperature of the specimen is measured with an electronic measuring device. Temperatures when crystals first appear, and then on warming disappear, are recorded.

FREEZING POINT:
D 5972
(Equivalent Test Method: IP 435)

This is an automated phase transition method used in the temperature range −80 to 20 °C. Results by this test method have a resolution of 0.1 °C, and have a precision better than that of Test Method D 2386. It also eliminates most of the operator time and judgment required in Test Method D 2386. In this test, a specimen is cooled at a rate of 15 ± 5 °C/min by a Peltier device while continuously being illuminated by a light source. The specimen is continuously monitored by an array of optical detectors for the first formation of solid hydrocarbon crystals. After that the specimen is warmed at the rate of 10 ± 0.5 °C/min until all crystals return to the liquid phase, and that temperature is also recorded.

TEST PRECISION

Test Method	Repeatability	Reproducibility
D 2386	0.8°C	2.3 °C
D 5901	0.46	1.43
D 5972	0.69	1.30

Bias of any of these three methods is not known. However, all three give equivalent results.

FREEZING POINT OF HIGH PURITY HYDROCARBONS:
D 1015

EXPLANATION

A knowledge of the purity of the hydrocarbons is often needed to help control their manufacture and to determine their suitability for use as reagent chemicals or for conversion to other chemical intermediates or finished products. The freezing point measured by this test method when used in conjunction with the physical constants for the hydrocarbons listed in Test Method D 1016, allows the determination of purity of these hydrocarbons.

TEST SUMMARY

The freezing points are precisely measured from the interpretation of time-temperature freezing or melting curves.

TEST PRECISION

Repeatability:	0.005°C
Reproducibility:	0.015°C

This test method has no bias.

FRETTING WEAR PROTECTION BY LUBRICATING GREASES:
D 4170

EXPLANATION

This test method is used to evaluate the property of lubricating greases to protect oscillating bearings from fretting wear. It is used for specification purposes, and differentiates between greases allowing low, medium, and high amounts of fretting wear under the prescribed test conditions. Test results do not necessarily correlate with results from other types of service.

TEST SUMMARY

The tester is operated with two ball thrust bearings, lubricated with the test grease. It is oscillated through an arc of 0.21 rad (12°) at a frequency of 30 Hz(1800 cpm) under a load of 2450 N(550 lbf) for 22 h at room temperature. Fretting wear is determined by measuring the mass loss of the bearing races.

TEST PRECISION

Repeatability:	$1.4\ X^{0.5}$
Reproducibility:	$3.0\ X^{0.5}$

Where X is the average mean mass losses of two tests in milligrams.

This procedure has no bias.

FRICTION AND WEAR PROPERTIES OF EXTREME PRESSURE LUBRICATING OILS:
D 6425
(Equivalent Test Method: DIN 51834)

EXPLANATION

This test method can be used to determine anti-wear properties and coefficient of friction of extreme pressure (EP) lubricating oils at selected temperatures and loads in applications for protecting against wear when subjected to high-frequency, linear oscillation motion. It has found application as a screening test for lubricants used in gear or cam/follower systems. No correlation is claimed between the results of this test method and field performance. This test method can also be used to determine the ability of a non-EP lubricating oil to protect against wear and its coefficient of friction under similar test conditions.

TEST SUMMARY

This test method is performed on an SRV test machine using a test ball oscillated at constant frequency and stroke amplitude and under controlled load against a test disk that has been moistened with the lubricant specimen. The platform to which the test disk is attached is held at a constant temperature. The friction force is measured by a piezoelectric device in the test disk assembly. Peak values of coefficient of friction are determined and recorded as a function of time. After a pre-set test period, the test machine and chart recorder are stopped and the wear scar on the ball is measured. If a profilometer is available, a trace of wear scar on the test disk can be used to obtain additional wear information.

TEST PRECISION

Measurement	Repeatability	Reproducibility
Average Ball Wear Scar Diameter (WSD)	0.1 mm	0.2 mm
Coefficient of friction	0.02	0.04

This test method has no bias.

FUEL SYSTEM ICING INHIBITORS IN AVIATION FUELS: D 5006

EXPLANATION

Diethylene Glycol Monomethyl Ether (DiEGME) is miscible with water and can be readily extracted from the fuel by contact with water during shipping and in storage. Methods are therefore needed to check the additive content in the fuel to ensure proper additive concentration in the aircraft. This test method is applicable to analyses performed in the field or a laboratory.

This test method covers a technique for measuring the DiEGME concentration in aviation fuels using HB and Brix scale refractometers. The extraction ratios are high enough that portable hand-held refractometers can be used, but not so high as to sacrifice accuracy or linearity, or both, in the 0.01–0.25 vol % range of interest.

TEST SUMMARY

A measured volume of fuel is extracted with a fixed ratio of water. The extraction procedure includes sufficient agitation and contacting time to ensure that equilibrium distributions are attained. With the HB refractometer, several drops of the water extract are placed on the prism face and the volume % DiEGME is read directly from a custom graduated scale printed on the reticule. When using the Brix refractometer, a temperature correction factor is first applied to the reading, multiplied by 2 and divided by 100 to calculate volume % DiEGME.

TEST PRECISION

Instrument	Bias	Repeatability	Reproducibility
HB Refractometer	−0.0018 vol %	0.009 vol %	0.018 vol %
Brix Refractometer	−0.0051 vol %	0.005 vol %	0.021 vol %

Accuracy and precision in the field can be lower than a similar test done under controlled laboratory conditions using a temperature controlled precision refractometer.

GAGE VAPOR PRESSURE OF LPG: D 1267

EXPLANATION

Information on the vapor pressure of LPG products under temperature conditions from 37.8 to 70°C is pertinent to selection of properly designed storage vessels, shipping containers, and customer utilization equipment to ensure safe handling of these products. For LPG, vapor pressure is an indirect measure of the most extreme low temperature conditions under which initial vaporization can be expected to occur. It can be considered a semi-quantitative measure of the amount of the most volatile material present in the product.

TEST SUMMARY

The test apparatus consisting of two interconnected chambers and equipped with a suitable pressure gage is purged with a portion of the sample which is then discarded. The apparatus is then filled completely with the portion of the sample to be tested. A 33.3 to 40 volume percent of the sample content of the apparatus is immediately withdrawn to provide adequate free space for product expansion. The apparatus is then immersed in a water bath maintained at the standard test temperature of 100°F or, optionally, at some higher test temperature up to and including a test temperature of 70°C. The observed gage pressure at equilibrium, after correcting for the gage error and correcting to a standard barometric pressure, is reported as the LPG vapor pressure at the selected test temperature.

TEST PRECISION

Repeatability:	12 kPa(1.8 psi)
Reproducibility:	19 kPa(2.8 psi)

This procedure has no bias because the value of vapor pressure is defined only in terms of this test method.

GASOLINE DILUENT IN USED ENGINE OILS DISTILLATION METHOD:
D 322

GENERAL:

Some fuel dilution of the engine oil may take place during normal operation. However, excessive fuel dilution is of concern in terms of possible performance problems. There are two test methods available for this work: one based on distillation, Test Method D 322 and the other based on gas chromatography, Test Method D 3525.

TEST SUMMARY

The sample, mixed with water, is placed in a glass still provided with a reflux condenser discharging into a graduated trap connected to the still. The contents of the still are brought to boiling by applying heat. The diluent in the sample is vaporized and then liquefied in the condenser. The diluent collects at the top of the trap and the excess water runs back to the still. The boiling is continued until all the diluent has been boiled out and recovered in the trap. The volume is recorded.

GASOLINE DILUENT IN USED ENGINE OILS GAS CHROMATOGRAPHY METHOD:
D 3525

TEST SUMMARY

This test method uses a gas chromatograph equipped with a flame ionization detector and a programmable oven. The use of other detectors and instrumentation has been reported. However, the precision statement applies only when the specified instrumentation is employed. *N*-tetradecane is used as the internal standard.

TEST PRECISION

Test Method	Repeatability	Reproducibility
D 322	0.6 volume %	1.4 volume %
D 3525	0.28	1.64

These are empirical test methods and have no bias.

GLYCERIN IN BIODIESEL METHYL ESTERS BY GC: D 6584

EXPLANATION

Free and bonded glycerin content reflects the quality of biodiesel. A high quantity of free glycerin may cause problems during storage, or in the fuel system, due to separation of the glycerin. A high total glycerin content can lead to injector fouling and may also contribute to the formation of deposits at injection nozzles, pistons, and valves.

This test method quantitatively determines free and total glycerin in b-100 methyl esters by gas chromatography. The range of detection for free glycerin is 0.005–05 mass %, and total glycerin from 0.05–0.5 mass %. This procedure is not applicable to vegetable oil methyl esters obtained from lauric oils, such as coconut oil and palmkernel oil.

TEST SUMMARY

The sample is silyated with N-methyl-N-trimethylsilytri-fluoracetamide and analyzed by gas chromatography. Calibration is achieved by the use of two internal standards and four reference materials. Mono-, di-, and triglycerides are determined comparing to monoolein, diolein, and triolein standards, respectively. Average conversion factors are applied to the mono-, di-, and triglycerides to calculate the bonded glycerin content of the sample.

TEST PRECISION

Component	Repeatability, mass %
Free Glycerin	0.001
Monoglycerides	0.021
Diglycerides	0.021
Triglycerides	0.032
Bound glycerin	0.008
Total glycerin	0.009

Reproducibility of the method has not yet been determined.

Since there is no accepted reference material suitable for determining bias for the procedure in this test method, bias cannot be determined.

GLYCOL ANTIFREEZE IN USED LUBRICATING OILS: D 2982

EXPLANATION

Leakage of glycol-base antifreeze into the crankcase is serious because the coolant tends to interfere with the lubricant and its ability to lubricate; it also promotes sludging, increases varnish deposit formation, and it will stay longer in the crankcase oil than water due to its higher boiling point. All this will lead to engine malfunction and possible seizure.

These tests are designed to detect glycol-base coolant even at low levels because early detection enable corrective measures to be taken to prevent leaking coolant from accumulating and seriously damaging the engine.

Another procedure, Test Method D 4291, is also used for the determination of ethylene glycol in used engine oils.

TEST SUMMARY

Two test procedures are available: one employing reagents in tablet form, the other employing laboratory shelf reagents. The ethylene glycol is extracted from the sample with an acid solution and oxidized to formaldehyde with periodic acid which is detected colorimetrically with decolorized fuchsin.

These tests methods will also detect other 1,2-glycols and carbohydrates that may be present. Hexylene- and methoxy-glycols, which are often used as gasoline anti-icing additives, do not interfere. Oil oxidation products present do not interfere. A modification is described for the tablet procedure for removing interferences due to carbohydrates such as sugars and sugar-containing substances that are sometimes used for sabotage purposes.

TEST PRECISION

These are qualitative pass-fail tests, and the normal precision estimates do not apply. The tablet procedure is sensitive to about 100 mg/kg and the shelf reagent procedure to about 30 mg/kg of ethylene glycol. Both procedures are adaptable to field kit use and commercial field testing kits are available.

GRAIN STABILITY OF CALCINED PETROLEUM COKE: D 6791

EXPLANATION

The grain stability of calcined petroleum coke determines the resistance to breakdown of +4 mm particles used in the manufacture of carbon anodes for use in the reduction process of aluminum. Cokes have to be relatively easy to grind for fines production but strong enough to withstand forming pressures and thermal stresses occurring when the anodes are used in the reduction process. This test method covers a laboratory vibration mill for the determination of grain stability of calcined coke for the manufacture of carbon products used in aluminum smelting. Calcined coke with poor mechanical strength may become degraded during mixing. Poor grain stability will affect the grain size and may result in poor quality of baked blocks.

TEST SUMMARY

A representative sample of calcined petroleum coke is dried and screened to a 4 mm to 8 mm fraction. The resultant sample is weighed so that two separate portions of 100.0 ± 0.5 g mass are obtained. The samples are placed into the laboratory mill and ground for a specific period of time. After grinding, the sample is screened and the mass of the +4 mm material is determined. The grain stability is the percent of the original materials remaining on the +4 mm sieve.

No material normally applied to the calcined petroleum coke is found to cause analytical interference with this procedure.

TEST PRECISION

Repeatability	2 %
Reproducibility	3 %

Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, no statement on bias can be made.

GRAVITY, API BY HYDROMETER METHOD: D 287

EXPLANATION

Accurate determination of the gravity of petroleum and its products is necessary for the conversion of measured volumes to volumes at the standard temperature of 60°F (15.56°C). Gravity is a factor governing the quality of crude oils. However, the gravity of a petroleum product is an uncertain indication of its quality. Correlated with other properties, gravity can be used to give approximate hydrocarbon composition and heat of combustion.

This test method covers the determination by means of a glass hydrometer of the API gravity of crude petroleum and petroleum products normally handled as liquids and having a Reid vapor pressure of 26 psi(180 kPa) or less. Gravities are determined at 60°F(15.56°C), or converted to values at 60°F, by means of standard tables. These tables are not applicable to nonhydrocarbons or essentially pure hydrocarbons such as the aromatics.

Also, see other gravity methods in the section on Density and Relative Density.

TEST SUMMARY

This test method is based on the principle that the gravity of a liquid varies directly with the depth of immersion of a body floating in it. The floating body, which is graduated by API gravity units in this method, is called an API hydrometer. The API gravity is read by observing the freely floating API hydrometer and noting the graduation nearest to the apparent intersection of the horizontal plane surface of the liquid with the vertical scale of the hydrometer, after temperature equilibrium has been reached. The temperature of the sample is read from a separate accurate ASTM thermometer in the sample or from the thermometer which is an integral part of the hydrometer (thermohydrometer).

TEST PRECISION

Repeatability:	0.2° API
Reproducibility:	0.5° API

EXISTENT GUM IN FUELS BY JET EVAPORATION:
D 381
(Equivalent Test Methods: IP 131, ISO 6246, DIN 51784, JIS K 2261, and AFNOR M07-004)

EXPLANATION

High gum can cause induction-system deposits and sticking of intake valves, and in most instances it can be assumed that low gum will ensure absence of induction-system difficulties. However, the test by itself is not correlative to induction system deposits. The test is applied to motor gasolines to measure the oxidation products formed in the sample prior to or during the comparatively mild conditions of the test procedure. Since many motor gasolines are purposely blended with nonvolatile oils or additives, the heptane extraction step is necessary to remove these from the evaporation residue so that the deleterious material, gum, may be determined. With respect to aviation turbine fuels, large quantities of gum are indicative of contamination of fuel by higher boiling oils or particulate matter and generally reflect poor handling practices in distribution downstream of the refinery.

TEST SUMMARY

A measured quantity of fuel is evaporated under controlled conditions of temperature and flow of air or steam. For aviation gasoline and aircraft turbine fuel, the resulting residue is weighed and reported. For motor gasoline, the residue is weighed before and after extracting it with *n*-heptane, and the results reported.

TEST PRECISION

Measurement	Repeatability	Reproducibility
Existent Gum (Aviation Gasoline)	$1.11+0.095\,X$	$2.09+0.126\,X$
Existent Gum (Aviation Turbine Fuel)	$0.5882+0.2490\,X$	$2.941+0.2794\,X$
Gum (unwashed)	$0.42\,X$	$1.333\,X$
Gum (washed)	$0.882+0.2746\,X$	$1.06+0.5567\,X$

Where X is the average of the results being compared.

These precision values were measured on motor gasoline samples only, and do not necessarily reflect the values obtainable on other product types.

The bias of this test method has not been determined.

HARDGROVE GRINDABILITY INDEX OF PETROLEUM COKE:
D 5003

EXPLANATION

Hardgrove Grindability Index (HGI) is a commonly done test on coal samples (See Test Method D 409). With the introduction of petroleum coke in the coal market, it has become necessary to extend this test to the former. The HGI is used to predict the ranking in industrial size mills used for crushing operations. The rankings are based on energy required and feed rate, or both. The HGI is also used to select raw petroleum cokes and coals that are compatible with each other when milled together in a blend so that segregation of the blend does not occur during particle size reduction.

TEST SUMMARY

The sample of petroleum coke is crushed to produce a high yield of particles passing a 1.19-mm (No. 16) sieve and retained on a 0.59-mm (No. 30) sieve. These particles are reduced in the hardgrove grindability machine according to the Test Method D 409. The quantity of particles retained on a 0.074-mm (No. 200) sieve is used to calculate the HGI of the sample.

TEST PRECISION

The precision data for this test is being developed.

This procedure has no bias. Both this test method and Test Method D 409 produce the same results on petroleum coke samples.

HEAT OF COMBUSTION OF PETROLEUM PRODUCTS NET HEAT OF COMBUSTION OF AVIATION FUELS:

D 1405

GENERAL

Heat of combustion is an important property particularly for the petroleum products that are used for burning, heating, or similar usage. A knowledge of this value is essential when considering the thermal efficiency of equipment for producing either power or heat. Heat of combustion per unit of mass fuel is a critical property of fuels intended for use in weight-limited crafts such as airplanes, surface effect vehicles, and hydrofoils. The range of such craft between refueling is a direct function of the heat of combustion and density of the fuel. There are several test methods to achieve this purpose, some of which are purely empirical in nature based only on calculations from other test data without actually physically measuring the heat of combustion.

This test method is purely empirical and applicable only to liquid hydrocarbon fuels derived from normal refining processes from crude oil. It is not applicable to pure hydrocarbons. It is not intended as a substitute for experimental measurements of heat of combustion. The calculations are based on the correlations between the net heat of combustion and the product of aniline point and API gravity. Correction for the sulfur content of the fuel must be carried out. This test method has a repeatability of 0.012 MJ/kg and a reproducibility of 0.035 MJ/kg.

No bias statement is made.

HEAT OF COMBUSTION OF AVIATION FUELS:

D 3338

Similar to Test Method D 1405, this is a purely empirical test based on correlation between the net heat of combustion and gravity, aromatic content, and the average volatility of the fuel. Correction for sulfur content of the fuel must be done. This test method has a repeatability of 0.021 MJ/kg and a reproducibility of 0.046 MJ/kg.

No bias statement is made.

HEAT OF COMBUSTION OF AVIATION FUELS:

D 4529

Similar to the other two methods, Test Methods D 1405 and D 3338, this is a purely empirical test method based on the correlation between the net heat of combustion and aniline point, density, and sulfur content. This test method has a repeatability of 0.012 MJ/kg and a reproducibility of 0.035 MJ/kg.

No bias statement is made.

HEAT OF COMBUSTION OF AVIATION FUELS:
D 6446

EXPLANATION

The net heat of combustion is a factor in the performance of all aviation fuels. For high performance weight-limited aircraft, the net heat of combustion per unit mass and the mass of fuel loaded determine the total safe range. The proper operation of the aircraft engine also requires a certain minimum net energy of combustion per unit volume of fuel delivered.

This test method is intended for use as a guide in cases where an experimental determination of heat of combustion is not available and cannot be made conveniently, and in which an estimate is considered satisfactory. It is not intended as a substitute for experimental measurements of heat of combustion which can be done by Test Methods D 240 and D 4809. This test method calculates the net heat of combustion from the fuel density, sulfur, and hydrogen content. This calculation is justifiable only when the fuel belongs to a well-defined class for which a relationship between these quantities has been derived from accurate experimental measurements on representative samples of that class. Even in this class, the possibility that the estimates can be in error by large amounts for individual fuels should be recognized.

TEST SUMMARY

The hydrogen content, density, and sulfur content of the sample are determined by experimental test methods and the net heat of combustion is calculated using the values obtained by these test methods based on reported correlations. The following test methods may be used for the actual analyses:

	ASTM Standards
Hydrogen	D 3701, D 4808, or D 5291
	D 1217, D 1298, D 4052, or Guide
Density at 15°C	D 1250
	D 129, D 1266, D 1552, D 2622, D 3120,
Sulfur	D 4294, or D 5453

TEST PRECISION

Repeatability:	0.05 MJ/kg
Reproducibility:	0.06 MJ/kg

Bias is not known.

HEAT OF COMBUSTION NET AND GROSS HEAT OF COMBUSTION OF BURNER AND DIESEL FUELS:
D 4868

Similar to the three test methods for aviation fuels, this is also an empirical test based on the correlation between heat of combustion and density, sulfur, water, and ash content of the fuel sample. The test is useful for use with burner and diesel fuels for which it is not usually critical to obtain very precise heat determinations. This test method is not applicable to pure hydrocarbons, nor is it intended as a substitute for experimental measurement of heat of combustion. This test method has a repeatability of 0.05 MJ/kg and a reproducibility of 0.15 MJ/kg.

No bias statement is made.

HEAT OF COMBUSTION HEATING VALUES OF LIQUIDS AND SOLIDS BY DIFFERENTIAL MACKEY TEST:
D 3523

EXPLANATION

The spontaneous heating value of a material is a measure of the ability of that material to undergo self-heating reactions while supported by a cellulosic or other fibrous material in air. It is an index of autoignition tendency of the substance under such conditions. This test method is not applicable to liquids on metal surfaces, on contaminated surfaces, or at pressures above atmospheric. The results by this test method are qualitative and should not be used to describe or appraise the fire-hazard or fire-risk of materials, products, or assemblies under actual fire conditions.

TEST SUMMARY

The sample is supported on surgical gauze and placed in a heated chamber that is open to air at the top. The temperature of this sample is compared to that of an equal reference quantity of surgical gauze contained in an identical chamber. Tests may be conducted for the durations of 4 to 72 h or longer.

PRECISION AND BIAS

No estimates are available since no round robins have been conducted given the complex and expensive nature of the equipment.

HEAT OF COMBUSTION OF LIQUID HYDROCARBON FUELS BY BOMB CALORIMETER:
D 240
(Equivalent Test Methods: IP 12 and AFNOR M07-030)

EXPLANATION

This test method is applicable to liquid hydrocarbon fuels ranging in volatility from that of light distillates to that of residual fuels. This test method is not as precise as Test Method D 4809 (see next).

TEST SUMMARY

Heat of combustion is determined by burning a weighed sample in an oxygen bomb calorimeter under controlled conditions.

TEST PRECISION

This test method has a repeatability of 0.13 MJ/kg and a reproducibility of 0.40 MJ/kg.

No bias statement is made.

HEAT OF COMBUSTION OF LIQUID HYDROCARBON FUELS BY BOMB CALORIMETER:
D 4809

EXPLANATION

This test method is specifically designed for use with aviation turbine fuels. It can be used for a wide range of volatile and nonvolatile materials where slightly greater difference in precision can be tolerated. Strict adherence to all test method details is essential to obtain good precision. The precision of this method is better than that by Test Method D 240 (see previous page).

TEST SUMMARY

A weighed sample is burned in an oxygen bomb calorimeter under controlled conditions. The temperature increase is measured by a temperature reading instrument which allows the precision of the method to be met. Either isoperibol or adiabatic calorimeters may be used.

TEST PRECISION

Samples	Repeatability, MJ/kg		Reproducibility, MJ/kg	
	Gross	Net	Gross	Net
All Fuels	0.097	0.096	0.228	0.324
Nonvolatile	0.096	0.099	0.239	0.234
Volatile	0.100	0.091	0.207	0.450

This test method has gross bias of 0.001 and a net bias of 0.089 MJ/kg.

HEAT OF COMBUSTION LIQUID HEAT CAPACITY OF PETROLEUM DISTILLATE FUELS:
D 2890

EXPLANATION

This is an empirical test based on calculations from Test Method D 86 distillation data. This test method is not applicable at temperatures less than 0°F(−18°C) and greater than 60°F(16°C) above the volumetric average boiling point of the fuel.

TEST SUMMARY

The Watson characterization factor, *K*, is obtained from a graphical correlation relating determined Test Method D 86 distillation data and *K*. The liquid heat capacity is obtained, either graphically or mathematically, from correlations relating calculated heat capacity, temperature at which it is being calculated, determined API gravity, and *K*.

TEST PRECISION

This test method has a repeatability of 0.01, and a reproducibility of 0.02 Btu/lb°F. The accuracy of this test method has been reported as within 4 % for straight-run petroleum fractions, and approximately 8 % for pure olefins.

No statement of bias is made.

HEAT OF COMBUSTION SPECIFIC HEAT OF AIRCRAFT TURBINE FUELS BY THERMAL ANALYSIS:
D 4816

EXPLANATION

This test method covers the determination of the specific heat of fuels by differential scanning calorimetry (DSC). These values are used in engineering calculations that relate to the manner in which a given system may react to thermal stress.

TEST SUMMARY

An empty, unsealed specimen pan is first heated over the desired temperature range while monitoring the heat supplied as a function of temperature, in a DSC instrument. Next, the program is repeated for the hermetically sealed pan containing a weighed test specimen of fuel. The specific heat is calculated from the difference between these two sets of measurements. A pure substance with known specific heat value is used as a calibrant.

TEST PRECISION

Since no round robins have been carried out, no statement of precision or bias can be made.

SEPARABILITY NUMBER OF HEAVY FUEL OILS BY OPTICAL SCANNING DEVICE:

D 7061

EXPLANATION

This procedure describes a rapid and sensitive method for estimating the stability reserve of an oil. The stability reserve is estimated in terms of a separability number, where a low value of separability number indicates that there is a stability reserve within the oil. When the separability number is between 0 to 5, the oil can be considered to have a high stability reserve and asphaltenes are not likely to flocculate. If the number is between 5 to 10, the stability reserve in the oil will be much lower. However, asphaltenes in this case are not likely to flocculate as long as the oil is not exposed to any worse conditions, such as storing, aging, and heating. If the separability number is above 10, the stability reserve of the oil is very low and asphaltenes will easily flocculate, or have already started to flocculate.

This test method can be used by refiners and users of oils, for which this test method is applicable, to estimate the stability reserves of their oils. This test method is not intended for predicting whether oils are compatible before mixing, but can be used for determining the separability number of already blended oils. However, oils that show a low separability number are more likely to be compatible with other oils than are oils with high separability numbers.

This test method covers the quantitative measurement, either in laboratory or in fields, of how easily asphaltene containing heavy oils diluted in toluene phase separate upon addition of heptane. The test method is limited to asphaltene containing heavy fuel oils, such as those in Specification D 396, Grades 4, 5, and 6, Specification D 975, Grade No. 4-D, and Specification D 2880, Grade Nos. 3-GT and 4-GT. Refinery fractions from which such blended fuels are made also fall within the scope of this test method.

TEST SUMMARY

Dilution of oil with toluene followed by addition of heptane causes asphaltenes to flocculate, and the oil to phase separate. The rate of the phase separation is determined by measuring the increase in transmission in the sample from the bottom of the test tube to the top (or a portion thereof) over time. The oils are first diluted with toluene in ratios that depend on the oil type. Two mL of the oil/toluene solution is mixed with 23 mL of heptane. Seven mL of this is transferred into a glass vial that is inserted into an optical scanning device. The change in light transmission through the glass vial containing the above mixture is recorded by scanning the vial vertically with the optical scanning device. One scan is run every 60 s for 15 min. An average of the transmittance is calculated from 1125 readings at 0.04 mm intervals along the glass vial, starting 10 mm above the bottom of the vial and continuing up to 55 mm for each scan. The separability number from 16 scans is calculated and reported.

TEST PRECISION

Following precision was obtained in a preliminary study:

Mean of Separability Number (%)	Standard Deviation (%)	95 % Confidence Interval (%)
0.3	0.02	0.01
6.9	0.6	0.4
17.0	0.4	0.3

Reproducibility of the test method is not yet determined.

Bias of this method cannot be determined since no accepted reference materials are available suitable for this test method.

HIGH TEMPERATURE DEPOSITS BY TEOST:

D 6335

EXPLANATION

The test method is designed to predict the high temperature deposit forming tendencies of an engine oil. This test method can be used to screen oil samples or as a quality assurance tool. It utilizes the thermo-oxidation engine oil simulation test (TEOST). The method is applicable over the range from 10 to 65 mg total deposits; although, operational experience has shown the test method to be applicable to engine oils having deposits over the range from 2 to 180 mg total deposit.

TEST SUMMARY

A sample of the engine oil at a temperature of 100°C that contains ferric napthenate and is in contact with nitrous oxide and moist air is pumped at a set flow rate past a tared depositor rod. The rod is resistively heated through twelve, 9.5 min temperature cycles that go from 200 to 480°C. When the twelve-cycle program is complete, the depositor rod rinsed of oil residue and dried and the gross rod mass obtained. The sample is flushed from the system and filtered through a tared filter. The mass of deposits on the rod plus the mass of deposits on the filter is the total deposit mass.

TEST PRECISION

Repeatability:	3.058 ($X^{0.33}$)
Reproducibility:	4.774 ($X^{0.33}$)

Where X is the mean of two values.

HIGH TEMPERATURE STABILITY OF DISTILLATE FUELS:
D 6468

EXPLANATION

This test method provides an indication of thermal oxidative stability of distillate fuels when heated to high temperatures that simulate those that may occur in some types of recirculating engine or burner fuel delivery systems. Results have not been substantially correlated to engine or burner operation. The test method can be useful for investigation of operational problems related to fuel thermal stability. When the test method is used to monitor manufacture or storage of fuels, changes in filter rating values can indicate a relative change in inherent stability. Storage stability predictions are more reliable when correlated to longer term storage tests, for example, Test Method D 4625, or other lower temperature, long-term tests

The test method uses a filter paper with a nominal porosity of 11 μm, which will not capture all of the sediment formed during aging but allows differentiation over a broad range. Reflectance ratings are also affected by the color of filterable insolubles, which may not correlate to the mass of the material filtered from the aged fuel sample. Therefore, no quantitative relationship exists between the pad rating and the gravimetric mass of filterable insolubles.

The test method is suitable to all No. 1 and No. 2 grades in Specifications D 396, D 975, D 2880, and D 3699, and for grades DMX and DMA in Specification D 2069. It is also suitable for similar fuels meeting other specifications. This test method is not suitable for fuels whose flash point, as determined by Test Methods D 56, D 93, or D 3828, is less than 38°C. This test method is also not suitable for fuels containing residual oils.

TEST SUMMARY

Two 50-mL volumes of filtered middle distillate fuel are aged for 90 or 180 min at 150°C in open tubes with air exposure. After aging and cooling, the fuel samples are filtered and the average amount of filterable insolubles is estimated by measuring the light reflectance of the filter pads. The 100 and 0 % extremes of the reflectance rating are defined by an unused filter pad and a commercial black standard, respectively.

TEST PRECISION

Aging Time	Repeatability	Reproducibility
90 min	22.42–0.2130 X	44.04–0.4281 X
180 min	22.5–50.2145 X	34.11–0.3034 X

Where X is the average of two results in percentage reflectance.

This test method bias cannot be determined since no accepted reference material is available.

HIGH TEMPERATURE UNIVERSAL OXIDATION TEST FOR TURBINE OILS:
D 6514

EXPLANATION

Degradation of fluid lubricants because of oxidation or thermal breakdown can result in fluid thickening or in the formation of acids or insoluble solids and render the fluid unfit for further use as a lubricant. This test method can be used for estimating the oxidation stability of oils. It can function as a formulation screening tool, specification requirement, quality control measurement, or as a means of estimating remaining service life. It shall be recognized, however, that correlation between results of this test method and the oxidation stability of an oil in field service can vary markedly with field service conditions and with various oils.

This test method is designed to complement Test Method D 5846 and is intended for evaluation of fluids which do not degrade significantly within a reasonable period of time at 135°C. It covers evaluating the oxidation of inhibited lubricants in the presence of air, copper, and iron metals. Although this test method was developed and used to evaluate the high temperature oxidation stability and deposit forming tendency of oils for steam and gas turbines, it has also been used for testing other lubricants made with mineral oil and synthetic basestocks for compressors, hydraulic pumps, and other applications, but these have not been tested in cooperative testing.

TEST SUMMARY

After determining the viscosity at 40°C and the acid number of a sample, a test specimen is stressed at 155°C for 96 hours. After cooling, the test specimen is vacuum filtered for the determination of total insolubles formed during the test. Total insolubles are reported as low, medium, or high. The viscosity and the acid number of the filtrate are determined and compared with the initial values to ascertain any increase in those values. Both the changes in acid number and the increase in viscosity at 40°C are reported. The glass cell in which the test specimen was stressed is rinsed with heptane and dried. Residual deposits are compared with ASTM Adjunct ADJD6514, and the results are reported.

TEST PRECISION

Parameter	Repeatability	Reproducibility
Sludge	$3.4\ X^{0.8}$	$7.4\ X^{0.8}$
Viscosity Change	$0.54(X+1)$	$1.59(X+1)$
Acid Number Change	$0.75(X+0.1)$	$1.57(X+0.1)$

Where X is the mean value.

This test method has no bias because the value of the oxidation life is defined only in terms of this test method.

HINDERED PHENOLIC AND AROMATIC AMINE ANTIOXIDANT CONTENT IN NON-ZINC TURBINE OILS BY LINEAR SWEEP VOLTAMMETRY:

D 6971

EXPLANATION

This test method covers the voltammetric determination of hindered phenol and aromatic amine antioxidants in new or used type non-zinc turbine oils in concentrations from 0.0075 mass % up to concentrations found in new oils by measuring the amount of current flow at a specified voltage in the produced voltammogram. This test method is not designed or intended to detect all of the antioxidant intermediates formed during the thermal and oxidative stressing of the oils, which are recognized as having some contribution to the remaining useful life of the used or in-service oil. Nor does it measure the overall stability of an oil, which is determined by the total contribution of all species present. Before making final judgment on the remaining useful life of the used oil, which might result in the replacement of the oil reservoir, it is advised to perform additional analytical techniques as in accordance with Test Methods D 6224, D 4378, and D 2272, having the capability of measuring remaining oxidative life of the used oil.

This test method is applicable to non-zinc type of turbine oils as defined by ISO 6743 Part 4, Table 1. These are refined mineral oils containing rust and oxidation inhibitors but not antiwear additives. The test is also suitable for manufacturing control and specification accceptance.

TEST SUMMARY

A measured quantity of sample is dispensed into a vial containing a measured quantity of acetone based electrolyte solution and a layer of sand. When the vial is shaken, the hindered phenol and aromatic amine antioxidants and other solution soluble oil components in the sample are extracted into the solution, and the remaining droplets suspended in the solution are agglomerated by the sand. The sand/droplet suspension is allowed to settle out and the hindered phenol and aromatic amine antioxidants dissolved in the solution are quantified by voltammetric analysis. The results are calculated and reported as mass % of antioxidant or as mmol of antioxidant per litre of sample for prepared and fresh oils, and as a % remaining antioxidant for used oils.

TEST PRECISION

Repeatability:	$1.5094(X+8.6662)^{0.4639}$
Reproducibility:	$3.0067(X+8.6662)^{0.4639}$

No information on bias can be presented since the result of this test is defined only in terms of this test method.

HINDERED PHENOLIC ANTIOXIDANT IN HL TURBINE OILS BY LINEAR SWEEP VOLTAMMETRY:

D 6810

EXPLANATION

This test method covers the voltammetric determination of hindered phenol antioxidants in new or used Type HL turbine oils in concentrations from 0.0075 wt. % up to concentrations found in new oils by measuring the amount of current flow at a specified voltage in the produced voltammogram. See Test Method D 6614 for further details.

TEST SUMMARY

A measured quantity of sample is dispensed into a vial containing a measured quantity of alcohol-based electrolyte solution and containing a layer of sand. When the vial is shaken, the hindered antioxidants and other solution soluble oil components present in the sample are extracted into the solution, and the remaining droplets suspended in the solution are agglomerated by the sand. The sand/droplet suspension is allowed to settle out and the hindered phenol antioxidants dissolved in the solution are quantified by voltammetric analysis. The results are calculated and reported as wt. % of antioxidant or as mmole of antioxidant per litre of sample for prepared and fresh oils, and as percent remaining antioxidant for used oils.

TEST PRECISION

Repeatability:	$0.7779\times(X+2)^{0.5}$ %
Reproducibility:	$1.830\times(X+2)^{0.5}$ %

Where X is the mean value.

The test method has no bias since the results of this test are defined only in terms of this test method.

HOMOGENITY AND MISCIBILITY OF ENGINE OILS:

D 6922

EXPLANATION

It is important that engine oils from different manufacturers be homogenous and miscible with each other, because operators of automotive engines often do not have prior knowledge of the manufacturer of the oil that is currently used in their application, and engine failure can occur if oils are combined that do not stay homogenous and function properly.

This test method covers the determination if an automotive engine oil is homogenous and will remain so, and if it is miscible with certain standard reference oils after being submitted to a prescribed cycle of temperature changes. This test method is very similar to the homogeneity and miscibility test described in FED-STD-791/3470.1.

TEST SUMMARY

Visual color determinations and observations are made on an undiluted test oil specimen, along with six blends of the same test oil that have been combined with specific reference oils. The pour point is then determined for the undiluted test oil specimen and the six blends. They are then allowed to warm to room temperature. Color determinations and observations are again made on the undiluted test oil specimen and six blends. They are then heated to 232°C, then allowed to cool to room temperature, and then stored at their pour point temperature for 18 to 24 h. All samples are then allowed to thaw and a series of color determinations and observations are made as they reach room temperature. All data are recorded on a report form.

TEST PRECISION

No information is presented about either the precision or the bias of this method since the test results are nonquantitative.

HYDROCARBON ANALYSIS OF PETROLEUM PRODUCTS:

GENERAL

The determination of hydrocarbon types—saturates, olefins, and aromatics—in petroleum fractions is important in characterizing the quality of petroleum fractions as gasoline blending components and as feeds to catalytic reforming processes, as products from thermal and catalytic cracking as blending components for motor and aviation fuels. This information is required in specifications such as Specification D 1655. There are a number of test methods for these analysis, all based on gas chromatography or gas chromatography-mass spectrometry. Each method is described in brief below. Also, see Test Method D 6293 for the determination of O-PONA hydrocarbons in fuels by gas chromatography, and Test Method D 6296 for the determination of olefins in the engine fuels by gas chromatography. Additionally, much more in-depth discussion of this subject can be found in the ASTM Manual on Hydrocarbon Analysis.

HYDROCARBON TYPES CHARACTERISTIC GROUPS IN OILS BY CLAY-GEL ABSORPTION CHROMATOGRAPHY: D 2007

EXPLANATION

This test method is used for classifying oil samples of initial boiling point of at least 200°C into the hydrocarbon types of polar compounds, aromatics, and saturates, and recovery of representative fractions of these types. This classification is used for specification purposes in rubber extendor and processing oils since the composition of the oil included in rubber compounds has a large effect on the characteristics and uses of the compounds. This test method is not directly applicable to oils of greater than 0.1 m % pentane insolubles. Such oils can be analyzed after removal of these materials, but precision is degraded.

TEST SUMMARY

The sample is diluted with solvent and charged to a glass percolation column containing clay at the top and silica gel plus clay in the bottom. The column is charged with *n*-pentane, and then the upper clay section is removed and washed further with *n*-pentane. Next it is charged with 50:50 toluene-acetone mixture for desorption. The gel column is desorbed with toluene. The solvents are completely removed from the recovered solvent fractions; the residues are weighed and calculated as saturates polar compound contents. Aromatics are calculated by difference, or measured after evaporation of the toluene fraction used for the desorption of the gel column.

TEST PRECISION

Analysis	Repeatability	Reproducibility
Saturate Content, m %	2.1	4.0
Aromatic Content, m %	2.3	3.3
Polar Content, m % of		
<1 %	0.24	0.4
between 1 to 5 %	0.81	0.3
of >5 %	1.2	1.8

This test method has no known bias.

HYDROCARBON TYPES AROMATICS AND NONAROMATICS FRACTIONS OF HIGH BOILING OILS BY EMULSION CHROMATOGRAPHY:

D 2549

EXPLANATION

The determination of compound types by mass spectrometry requires, in some instances, a preliminary separation of the petroleum sample into representative aromatics and nonaromatics fractions, such as in Test Methods D 2425, D 2786, and D 3239. This test method provides a suitable separation technique for this application. This test method is suitable for hydrocarbon mixtures boiling between 232 and 538°C. Alternative procedures are provided for 2 or 10 g of hydrocarbon mixture. Some components may not be eluted from the chromatographic columns for some type of samples under the conditions used in this test.

Test Method D 2007 is an alternative method of separating high-boiling oils into polar compounds, aromatics, and saturate fractions.

TEST SUMMARY

A sample is charged to the top of a glass chromatographic column packed with activated bauxite and silica gel. *N*-Pentane is used to elute the nonaromatics from the column. When all of them are eluted, the aromatic fraction is eluted with diethyl ether, chloroform, and ethyl alcohol. The solvents are completely removed by evaporation, the residues are weighed and calculated as the aromatics and nonaromatics fractions of the sample.

TEST PRECISION

Sample Weight, g	Repeatability, Weight %,	Reproducibility, Weight %
2	1.4	1.5
10	0.4	1.6

The bias of this test method has not been determined.

HYDROCARBON TYPES BY FLUORESCENT INDICATOR ADSORPTION:

D 1319

(Equivalent Test Methods: IP 156, ISO 3837, DIN 51791, JIS K 2536, and AFNOR M07-024

EXPLANATION

This test method is intended for use with full boiling range products. It determines hydrocarbon types in the range of 5 to 99 volume % aromatics, 0.3 to 55 volume % olefins, and 1 to 95 volume % saturates in petroleum fractions that distill below 315°C.

TEST SUMMARY

A small amount of sample is introduced into a special glass adsorption column packed with activated silica gel. A small layer of silica gel contains a mixture of fluorescent dyes. When all of the sample has been adsorbed on the gel, alcohol is added to desorb the sample down the column. The hydrocarbons are separated according to their affinities into three types. The fluorescent dyes also react selectively with the hydrocarbon types, and make the boundary zones visible under ultraviolet light. The volume percentage of each hydrocarbon type is calculated from the length of each zone in the column.

TEST PRECISION[1]

The precision statement has been determined with unleaded fuels that do not contain oxygenates. It may or may not apply to other types of gasolines. See the repeatability and reproducibility in the following tables.

[1] These tables are Tables 2 and 3 from Test Method D 1319.

Reproducibility and Repeatability—Oxygenate Free Samples.

	Volume %		
	Level	Repeatability	Reproducibility
Aromatics	5	0.7	1.5
	15	1.2	2.5
	25	1.4	3.0
	35	1.5	3.3
	45	1.6	3.5
	50	1.6	3.5
	55	1.6	3.5
	65	1.5	3.3
	75	1.4	3.0
	85	1.2	2.5
	95	0.7	1.5
	99	0.3	0.7
Olefins	1	0.4	1.7
	3	0.7	2.9
	5	0.9	3.7
	10	1.2	5.1
	15	1.5	6.1
	20	1.6	6.8
	25	1.8	7.4
	30	1.9	7.8
	35	2.0	8.2
	40	2.0	8.4
	45	2.0	8.5
	50	2.1	8.6
	55	2.0	8.5
Saturates	1	0.3	1.1
	5	0.8	2.4
	15	1.2	4.0
	25	1.5	4.8
	35	1.7	5.3
	45	1.7	5.6
	50	1.7	5.6
	55	1.7	5.6
	65	1.7	5.3
	75	1.5	4.8
	85	1.2	4.0
	95	0.3	2.4

Reproducibility and Repeatability for Oxygenate Containing Samples.

	Range	Repeatability, Volume %	Reproducibility
Aromatics	13–40	1.3	3.7
Olefins	4–33	$0.2578X^{0.6}$	$0.8185X^{0.6}$
Saturates	45–68	1.5	4.2

Where X = the volume % of olefins.

The bias of this test method has not been determined.

HYDROCARBON TYPES IN GASOLINE BY GAS CHROMATOGRAPHY:

D 2427

EXPLANATION

This test method provides information on C_2 through C_5 carbon paraffins and mono-olefins in gasolines. It does not determine cyclic olefins, diolefins, or acetylenes. These are usually minor components in finished gasolines. The samples should not contain significant amounts of material boiling lower than ethylene.

TEST SUMMARY

The sample is injected into a gas-liquid partition column. The components are separated as they pass through the column with an inert carrier gas and their presence in the effluent is detected and recorded on a chromatograph. Sample composition is determined from the chromatogram by comparing peak areas with those obtained using known amounts of calibration standards or a synthetic blend.

TEST PRECISION[1]

Following precision applies for total C_5 and lighter olefins, and total C_5 and lighter hydrocarbons:

	Range, %	Repeatability	Reproducibility
Total C_5 and lighter Olefins	6.5 to 12	0.4	2.5
Total C_5 and lighter Hydrocarbons	11 to 20	0.6	3.3

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[1] This table is Table 1 in Test Method D 2427.

For individual components' precision see table below.

Precision Data.

Component	Concentration, %	Repeatability	Reproducibility
Olefins:			
Propylene	0.2 to 0.8	0.1	0.3
Butene-1 plus isobutylene	1.5 to 1.9	0.1	0.5
trans-Butene-2	1.0 to 1.3	0.1	0.4
cis-Butene-2	0.8 to 1.0	0.1	0.3
Pentene-1	0.2 to 0.5	0.1	0.2
2-Methylbutene-1	0.9 to 1.5	0.2	0.3
trans-Pentene-2	1.0 to 1.5	0.1	0.3
cis-Pentene-2	0.5 to 0.8	0.1	0.3
2-Methylbutene-2	2.5 to 3.2	0.3	0.8
Paraffins:			
Propane	0.1 to 0.5	0.1	0.2
Isobutane	0.5 to 2.0	0.1	0.3
n-Butane	0.3 to 0.9	0.1	0.2
Isopentane	2.8 to 4.0	0.3	0.4
n-Pentane	0.8 to 1.8	0.1	0.2

The bias of this test method is not known.

HYDROCARBON TYPES IN ETHYLENE BY GAS CHROMATOGRAPHY:

D 2505

EXPLANATION

High purity ethylene is required as a feedstock for some manufacturing processes, and the presence of trace amounts of carbon dioxide and some hydrocarbons can have a deleterious effect. This test method can be used for setting specifications or for use as internal quality control. This test method is applicable over the range of impurities from 1 to 500 ppm by volume. It covers the determination of carbon dioxide, methane, ethane, acetylene, and other hydrocarbons.

TEST SUMMARY

The sample is separated in a gas chromatograph utilizing four different packed chromatographic columns with helium as the carrier gas. Methane and ethane are determined using a silica gel column, propylene and heavier hydrocarbons with a hexamethylphosphoramide column, acetylene in a series of hexadecane and a squalane column, and carbon dioxide with an activated charcoal impregnated with a solution of silver nitrate. Calibrations are done using standard samples containing known amounts of these compounds.

TEST PRECISION

Component	Range	Repeatability	Reproducibility
Ethylene	99.80–99.99 mol %	0.006 mol %	0.1 mol %
Methane	1–150 ppmV	3 ppmV	34 ppmV
Ethane	1–1500 ppmV	43 ppmV	72 ppmV
Propylene	1–15 ppmV	3 ppmV	290 ppmV
Propane	1–15 ppmV	2 ppmV	11 ppmV
Acetylene	1–20 ppmV	1 ppmV	7 ppmV
Carbon dioxide	1–10 ppmV	1 ppmV	6 ppmV
			4 ppmV

The bias of this method has not been determined.

HYDROCARBON TYPES IN ENGINE FUELS BY GAS CHROMATOGRAPHY:

D 6839

EXPLANATION

A knowledge of spark ignition engine fuel composition is needed for regulatory compliance, process control, and quality assurance. A quantitative determination of olefins and other hydrocarbon types in such fuels is required to comply with government regulations. This test method is not applicable to M85 and E85 fuels which contain 85 % of methanol and ethanol, respectively. This test method quantitatively determines saturates, olefins, aromatics, and oxygenates in spark ignition engine fuels by multidimensional gas chromatography. Each hydrocarbon type can be reported either by carbon number or as a total. The method is applicable to fuels with total aromatic content up to 50 %, total olefinic content up to 30 %, and oxygen compounds up to 15 %, all v/v. This method is not intended to determine individual hydrocarbon components except benzene.

TEST SUMMARY

A representative sample is introduced into a computer controlled gas chromatographic (GC) system consisting of switching valves, columns, and an olefin hydrogenation catalyst, all operating at various temperatures. The valves are actuated at predetermined times to direct portions of the sample to appropriate columns and traps. As the analysis proceeds, the columns separate these sample portions sequentially into groups of different hydrocarbon types that elute to a flame ionization detector. The mass concentration of each detected compound or hydrocarbon group is determined by application of response factors to the areas of detected peaks followed by normalization to 100 %. The liquid volume concentration of each detected compound or hydrocarbon group is determined by application of density factors to the calculated mass concentration of the detected peaks followed by normalization to 100 %.

INTERFERENCES—Oxygenates as specified in Test Method D 4815 do not interfere with hydrocarbons. Some types of sulfur containing compounds are irreversibly adsorbed in the olefin trap reducing its capacity to retain olefins. Sulfur containing compounds are also adsorbed in the alcohol and ether-alcohol-aromatic trap. However, a variety of spark ignition engine fuels have been analyzed without significant performance deterioration of these traps. Commercial dyes used to distinguish between grades and types of these fuels have been found not to interfere with the test method.

TEST PRECISION[1]

Since no suitable reference standards are available for this analysis, no statement regarding the bias of this method can be made.

NOTE—The reporting unit for the covered range is in liquid volume percent except for the level of oxygen, which is in weight percent

Component or Group	Repeatability	Reproducibility	Covered Range
Aromatics	$0.012\ (10+X)$	$0.036\cdot(10+X)$	20–45 v/v
Olefins	$0.13\cdot X^{0.46}$	$0.72\cdot X^{0.46}$	0–28 v/v
Saturates	0.5	1.6	25–80 v/v
Oxygen	0.02	0.10	0.25–1.8 m/m
Benzene	$0.019\cdot X^{1.6}$	$0.053\cdot X^{1.6}$	0.5–1.6 v/v
MTBE	0.14	0.37	10 v/v
Ethanol	0.06	0.37	0.5–4 v/v
ETBE	0.09	0.67	10 v/v
TAME	0.07	0.71	4.5 v/v

[1] This table is Table 1 in Test Method D 6839-02.

HYDROCARBON TYPES BY MASS SPECTROMETRY:

D 2786

EXPLANATION

This test method covers the determination of seven saturated hydrocarbon types and one aromatic type using high ionizing voltage mass spectrometry. It covers the analysis of saturate petroleum fractions with average carbon numbers of 16 through 32. Samples must be nonolefinic and must contain less than 5 volume % monoaromatics. When used together with Test Method D 3239 (see following), this test method provides a detailed analysis of the hydrocarbon composition of such materials.

TEST SUMMARY

The relative abundance of these hydrocarbons in petroleum saturate fractions is determined by mass spectrometry using a summation of mass fragment groups most characteristic of each molecular type. Calculations are carried out by the use of inverted matrices that are specific for any average carbon number. The saturate fraction necessary for this test is obtained by liquid elution chromatography Test Method D 2549.

TEST PRECISION

Based on a cooperative study, the following precisions were obtained.

Rings	Volume %	Repeatability	Reproducibility
0	10.5	1.5	5.4
1	17.3	1.8	5.0
2	15.8	0.5	4.9
3	16.7	0.8	3.2
4	30.1	2.0	11.0
5	6.8	0.9	4.3
6	2.8	0.7	3.5

This test method has no bias.

HYDROCARBON TYPES AROMATICS TYPES BY MASS SPECTROMETRY:

D 3239

EXPLANATION

This test method covers the determination by high ionizing voltage, low resolution mass spectrometry of 18 aromatic hydrocarbon types and three aromatic thiophenotypes in straight run aromatic petroleum fractions boiling within the range from 205 to 540°C(400 to 1000°F). Samples must be nonolefinic, must not contain more than 1 mass % of total sulfur, and must not contain more than 5 % nonaromatic hydrocarbons.

TEST SUMMARY

The relative abundances of seven classes of aromatics in petroleum fractions are determined by mass spectrometry using a summation of peaks most characteristic of each class. Calculations are carried out by the use of inverted matrix derived from the published spectra of pure aromatic compounds. The aromatic fraction needed for this analysis is obtained by using liquid elution chromatography by Test Method D 2549.

TEST PRECISION[1]

Based on the interlaboratory study, the following precision estimates were obtained.

Precision Summary Based on Cooperative Data.

	Vol %	σ_r	σ_R	r	R
Alkylbenzenes	13.7	0.3	1.0	1.2	3.0
Naphthenebenzenes	13.3	0.1	1.1	0.5	3.3
Dinaphthenebenzenes	13.7	0.2	0.4	0.9	1.1
Naphthalenes	6.7	0.2	0.8	0.9	2.3
Acenaphthenes/dibenzofurans	9.0	0.1	0.2	0.5	0.5
Ruorens	10.7	0.1	0.2	0.3	0.6
Phenanthrenes	8.6	0.1	0.3	0.2	1.0
Naphthenephenanthrenes	4.5	0.2	0.4	0.7	1.2
Pyrenes	5.7	0.1	0.5	0.3	1.6
Chrysenes	2.8	0.2	0.4	0.5	1.1
Perylenes	1.7	0.1	0.2	0.3	0.6
Dibenzanthracenes	0.4	0.1	0.1	0.2	0.4
Benzothiophenes	1.0	0.2	0.4	0.8	1.1
Dibenzothiophenes	1.5	0.1	0.3	0.3	0.8
Naphthabenzothiophenes	0.5	0.1	0.3	0.3	1.0
Class II Unidentified	0.4	0.1	0.4	0.3	1.1
Class III Unidentified	0.6	0.1	0.4	0.4	1.2

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[1] This table is Table 5 from Test Method D 3239.

Class IV Unidentified	4.1	0.2	0.5	0.6	1.6
Class V Unidentified	0.5	0.1	0.3	0.5	0.8
Class VI Unidentified	0.2	0.1	0.1	0.3	0.4
Class VII Unidentified	0.4	0.2	0.2	0.5	0.7

σ_r = repeatability standard deviation

σ_R = reproducibility standard deviation

r = repeatability

R = reproducibility

The bias of this test method is not known.

HYDROCARBON TYPES AROMATIC HYDROCARBON TYPES IN AVIATION FUELS AND PETROLEUM DISTILLATES:

D 6379

(Equivalent Test Method: IP 436)

EXPLANATION

Accurate quantitative information on aromatic hydrocarbon types can be useful in determining the effects of petroleum processes on production of various finished fuels, and for indicating the quality of fuels and for assessing the relative combustion properties of finished fuels. This test method is intended to be used as one of the several possible alternative instrumental test methods that are used for quantitative determination of hydrocarbon types in fuels. This does not imply that a correlation necessarily exists between this and any other test method intended to give this information.

This test method determines mono- and di-aromatic hydrocarbon contents in aviation kerosenes and petroleum distillates boiling in the range from 50 to 300°C, such as Jet A or Jet A-1 fuels. This test method is calibrated for distillates containing from 10 to 25 % m/m monoaromatic, and from 0 to 7 % m/m di-aromatic hydrocarbons. Compounds containing sulfur, nitrogen, and oxygen are possible interferents. Mono-alkenes do not interfere, but conjugated di- and poly-alkenes, if present, are possible interferents.

TEST SUMMARY

The sample is diluted 1:1 with a solvent such as heptane, and a fixed volume of this solution is injected into a high performance liquid chromatograph fitted with a polar column. The column selectively separates the aromatic from the nonaromatic hydrocarbons into distinct bands in accordance with their ring structure, that is, MAHs and DAHs. The column is connected to a refractive index detector that detects the components as they elute from the column. The electronic signal from the detector is continually monitored by a data processor. The peak areas of the sample aromatics are compared with those obtained from previously run calibration standards to calculate the percent m/m MAHs and DAHs in the sample. Their sum is reported as the total aromatic content of the sample.

TEST PRECISION

	Range (% Mass)	Repeatability	Reproducibility
Di-aromatics	0.10–6.64	$0.337X^{0.333}$	$0.514X^{0.333}$
Mono-aromatics	10.5–24.1	$0.129X^{0.667}$	$0.261X^{0.667}$

Where X is the average of results being compared.

There is no bias.

AROMATIC HYDROCARBON TYPES IN MIDDLE DISTILLATES BY HPLC WITH RI DETECTION: **D 6591**

(Equivalent Test Methods: IP 391)

EXPLANATION

The aromatic hydrocarbon content of motor diesel fuel is a factor that can affect exhaust emissions and fuel combustion characteristics, as measured by cetane number. Both U.S. EPA and California Air Resources Board limits the aromatic hydrocarbon content of diesel fuels. This test method is applicable to materials in the same boiling range as motor diesel fuels and is unaffected by fuel coloration.

This test method is intended to be used as one of several possible alternative instrumental test methods meant for quantitative determination of hydrocarbon types in fuels. It does not, however, imply that a correlation necessarily exists between this and any other test method intended to give this information.

This test method covers a high performance liquid chromatographic (HPLC) test method for the determination of monoaromatic, di-aromatic, and poly-aromatic hydrocarbon contents in diesel fuels and petroleum distillates boiling in the range from 150 to 400°C. The total aromatic content in % m/m is calculated from the sum of the individual aromatic hydrocarbon types. Aviation fuels and petroleum distillates with a boiling point range from 50 to 300°C are not determined by this test method and should be analyzed by Test Method D 6379, or other suitable test methods.

This test method is calibrated for distillates containing from 4 to 40 % (m/m) mono-aromatic hydrocarbons, 0 to 20 % (m/m) di-aromatic hydrocarbons, 0 to 6 % (m/m) polycylic aromatic hydrocarbons, and 4 to 65 % (m/m) total aromatic hydrocarbons. Compounds containing sulfur, nitrogen, and oxygen are possible interferents. Mono-alkenes do not interfere, but conjugated di- and poly-alkenes, if present, are possible interferents.

TEST SUMMARY

A known mass of the sample is diluted in the mobile phase, and a fixed volume of this solution is injected into an HPLC instrument fitted with a polar column. This column has little affinity for the nonaromatic hydrocarbons while exhibiting a pronounced selectivity for aromatic hydrocarbons. As a result of this selectivity, the aromatic hydrocarbons are separated from the nonaromatic hydrocarbons into distinct bands in accordance with their ring structure, that is, MAHs, DAHs, and PAHs. At a predetermined time after the elution of DAHs, the column is back-flushed to elute the PAHs as a single sharp band. The column is connected to a refractive index (RI) detector that detects the components as they elute from the column. The electronic signal from the detector is continually monitored by a data processor. The amplitudes of the signals (peak areas) from the sample aromatics are compared with those obtained from previously run calibration standards in order to calculate % (m/m) of MAHs, DAHs, and PAHs in the sample. The sum of these is reported as total aromatic content (% m/m) of the sample.

TEST PRECISION

No information can be presented on the bias of this procedure because no material having an accepted reference value is available.

Precision Values.

Aromatic Type	Range % (m/m)	Repeatability	Reproducibility
Mono-aromatic hydrocarbons	4–40	$0.026(X^{a}+14.7)$	$0.063(X+17.3)$
Di-aromatic hydrocarbons	0–20	$0.10(X+2.6)$	$0.32(X+1.8)$
Poly-aromatic hydrocarbons	0–6	$0.12(X+0.6)$	$0.64(X+0.3)$
Total aromatic hydrocarbons	4–65	$0.036(X+1.5)$	$0.116(X+6.3)$

[a] X = Average of results being compared.

HYDROCARBON TYPES BY MASS SPECTROMETRY: D 2425

EXPLANATION

This test method covers an analytical scheme using the mass spectrometer to determine the hydrocarbon types present in virgin middle distillates in the boiling range 204 to 343°C (400 to 650°F), 5 to 95 volume % as determined by Test Method D 86. Eleven hydrocarbon types with average carbon number value of paraffins from C_{10} to C_{18} can be analyzed. If nonhydrocarbon types such as sulfur and nitrogen containing compounds are present to any larger extent they will interfere with the spectral peaks used for the hydrocarbon-type calculation.

TEST SUMMARY

Samples are separated into saturate and aromatic fractions by Test Method D 2549, and each fraction is analyzed by mass spectrometry. The analysis is based on the summation of characteristic of mass fragments to determine the concentration of hydrocarbon types. The average carbon numbers of the hydrocarbon types are estimated from the spectral data. Calculations are made from the calibration data dependent upon the average carbon number of hydrocarbon type.

TEST PRECISION[1]

Based on an interlaboratory study, the following precision was obtained.

Precision of Method.

Compound	Concentration Mass, %	Repeatability	Reproducibility
Saturate Fraction:			
Paraffins	40 to 50	0.5	4.0
Monocycloparaffins	18 to 25	1.1	5.2
Dicycloparaffins	6 to 12	0.7	4.4
Tricycloparaffins	1 to 5	0.3	2.0
Alkylbenzenes	0 to 3	0.2	0.3
Aromatic Fraction:			
Paraffins	0 to 2	0.4	0.6
Cycloparaffins	0 to 2	0.5	0.9
Alkylbenzenes	3 to 8	0.3	1.4
Indan and/or tetralins	2 to 5	0.3	0.5
C_nH2n–10	0 to 4	0.3	0.7
Naphthalenes	3 to 8	0.3	1.0
C_nH2n–14	0 to 3	0.1	0.9
C_nH2n–16	0 to 3	0.3	0.7
C_nH2n–18	0 to 3	0.1	0.4

The bias of this test method is not known.

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[1] This table is Table 6 from Test Method D 2425.

HYDROCARBON TYPES IN GASOLINE BY MASS SPECTROMETRY: D 2789

EXPLANATION

This test method covers the determination by mass spectrometry of total paraffins, monocycloparaffins, dicycloparaffins, alkylbenzenes, indans, or tetralins, or both, and napthalenes in gasoline having an olefin content of less than 3 volume % and a 95 % distillation point of less than 210°C as determined by Test Method D 86. This method has not been tested on gasolines containing oxygenates such as alcohols and ethers.

TEST SUMMARY

Samples are analyzed by mass spectrometry, based on the summation of characteristic mass fragments, to determine the concentration of the hydrocarbon types. The average number of carbon atoms of the sample is estimated from spectral data. Calculations are made from calibration data which are dependent upon the average number of carbon atoms of the sample.

TEST PRECISION[1]

Based on an interlaboratory study, the following precision was obtained.

Precision Data for Cooperative Samples.

Type	Naphtha					Reformat				
	Volume Percent	σ_r	σ_R	r	R	Volume Percent	σ_r	σ_R	r	R
Paraffins	52.6	0.3	1.7	1.0	5.3	34.2	0.4	1.7	1.3	5.3
Monocycloparaffins	34.6	0.2	1.8	0.7	5.6	4.0	0.1	0.6	0.3	1.8
Dicycloparaffins	5.2	0.1	0.5	0.4	1.7	0.1	0.0	0.1	0.0	0.2
Alkylbenzenes	6.3	0.1	0.4	0.4	1.4	56.6	0.2	2.1	0.6	6.8
Indans and tetralins	0.9	0.1	0.1	0.1	0.4	2.2	0.1	0.5	0.3	1.6
Naphthalenes	0.3	0.0	0.1	0.1	0.4	3.0	0.1	0.8	0.3	2.6

σ_r = repeatability standard deviation.

σ_R = reproducibility standard deviation.

r = repeatability.

R = reproducibility.

The bias for this test method has not been determined.

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[1] This table is Table 5 from Test Method D 2789.

HYDROCARBON TYPES BY MULTIDIMENSIONAL GC: D 5443

EXPLANATION

This test method is applicable to hydrocarbon mixtures including virgin, catalytically or thermally converted, alkylated, and blended napthas. The method provides for the determination of paraffins, napthenes, and aromatics by carbon number in low olefinic hydrocarbon streams having final boiling points of 200°C or less. Hydrocarbons in the boiling range 200 to 270°C are reported as a single group. Aromatics boiling at C_9 and above are reported as a single aromatic group. Chemicals of a nonhydrocarbon composition may elute within the hydrocarbon groups depending on their polarity, boiling point, and molecular size. This group includes ethers and alcohols.

TEST SUMMARY

A sample is injected into a gas chromatographic system containing a series of columns and switching values. First, a polar column retains polar aromatic compounds, binapthenes, and high boiling paraffins and napthenes. The eluant from this column goes through a platinum column which hydrogenates olefins, and then to a molecular sieve column which performs a carbon number separation based on the molecular structure, that is, napthenes and paraffins. The fraction remaining on the polar column is further divided into three separate fractions that are then separated on a nonpolar column by boiling point. Eluting compounds are detected by a flame ionization detector. The mass concentration of each group is determined by the multiplication of detected peak areas by flame ionization detector response factors and normalized to 100 %.

TEST PRECISION[1]

Based on an interlaboratory study, the following precision was obtained.

[1] This table is Table 9 from Test Method D 5443.

Repeatability and Reproducibility for Selected Naphtha Components and Groups of Components

Component or Group	Repeatability[a]	Reproducibility[a]
Benzene	$0.066(x)^{0.20}$	$0.20(x)^{0.20}$
Toluene	$0.51(x)^{0.67}$	$0.22(x)^{0.67}$
C_8 Aromatics	$0.041(x)$	$0.17(x)$
C_{9^+} Aromatics	$0.92(x)^{0.50}$	$0.50(x)^{0.50}$
C_7 Paraffins	0.16 %	0.61 %
C_8 Paraffins	$0.066(x)^{0.50}$	$0.18(x)^{0.50}$
C_9 Paraffins	$0.098(x)^{0.50}$	$0.17(x)^{0.50}$
C_6 Naphthenes	$0.046(x)^{0.50}$	$0.11(x)^{0.50}$
C_7 Naphthenes	$0.14(x)$	$0.33(x)$
C_8 Naphthenes	$0.067(x)^{0.33}$	$0.13(x)^{0.33}$
Total paraffins	$0.064(x)^{0.50}$	$0.17(x)^{0.50}$
Total naphthenes	$0.059(x)^{0.50}$	$0.11(x)^{0.50}$
Total aromatics	$0.077(x)^{0.50}$	$0.28(x)^{0.50}$

[a] (x) Refers to the mass percent of the component or group of components found.

The bias of this test method has not been determined.

HYDROGEN CONTENT OF FUELS

GENERAL

Hydrogen content of petroleum products represents a fundamental quality parameter that has been correlated with many of the performance characteristics of that product. Many times hydrogen content is required to correct gross heat of combustion to net heat of combustion which is used in aircraft calculations because all combustion products are in the gaseous state.

There are five methods available for this determination, which are described here. One of them, Test Method D 5291, is described under the nitrogen determination section.

HYDROGEN CONTENT OF AVIATION FUELS: D 3343

This is an empirical method based on calculations and is applicable to aviation gasolines and aircraft turbine and jet engine fuels.

TEST SUMMARY

The calculations are done based on the correlationship between hydrogen content of a fuel and its distillation range, API gravity, and its distillation range. The correlation is not accurate when the values of the variables used in the equation are within one standard deviation of the mean, but is useful up to two standard deviations of the mean.

TEST PRECISION

This test method has a repeatability of 0.03 % and a reproducibility of 0.10 %. The standard error of estimate for hydrogen content of all fuels is 0.20 % and for aviation type fuels is 0.16 %.

HYDROGEN CONTENT OF AVIATION TURBINE FUELS BY NUCLEAR MAGNETIC RESONANCE (NMR):
D 3701

This test method provides a quick, simple, and precise alternative method to that using empirical correlationships.

TEST SUMMARY

A sample of the fuel is compared in a continuous wave, low-resolution, nuclear magnetic resonance spectrometer with a reference standard sample of pure hydrocarbon. The method is specifically based on Newport analyzers.

TEST PRECISION

This test method has a repeatability of 0.09 mass % and a reproducibility of 0.11 mass %. It has been reported that the hydrogen content determined by this test method is biased high with respect to the expected value for pure known materials.

HYDROGEN CONTENT OF PETROLEUM PRODUCTS BY NMR:
D 4808

These test methods are a precise and simple alternative to other methods such as combustion or empirical methods for hydrogen determination in fuels. They use a continuous wave, low-resolution nuclear magnetic resonance spectrometer. Three test methods are included here to take into account special characteristics of different petroleum products: light distillates, middle distillates and gas oils, and residua.

TEST SUMMARY

See description under Test Method D 3701.

TEST PRECISION

Test Method	Range	Repeatability	Reproducibility
A	Light Distillates	$0.22(X^{0.25})$	$0.72(X^{0.25})$
B	Middle Distillates and Gas Oils	$0.0015(X^2)$	$0.0032(X^2)$
C	Residua	$33.3(X^{-2})$	$70.3(X^{-2})$

Where X is the sample mean.

A 1985 research report indicated that these test methods are not biased with respect to data obtained by the combustion techniques. A 1977 research report, however, indicates that Test Method A (same as Test Method D 3701) is biased high with respect to the expected value of pure known compounds.

HYDROGEN BY NMR:
D 7171

EXPLANATION

Hydrogen content represents a fundamental quality of a petroleum distillate that has been correlated with many of the performance characteristics of that product. Combustion properties of gas turbine fuels are related primarily to hydrogen content. As hydrogen content of these fuels decreases, soot deposits, exhaust smoke, and thermal radiation increase. Soot deposits and thermal radiation can increase to the point that combustor liner burnout will occur. Hydrogen content is a procurement requirement for military fuels Jp-5 specified in MIL-DTL-5624U, JP-8 specified in MIL-DTL-83133E, and

Naval Distillate specified in MIL-PRF-16884K. This test method provides a simple and precise alternative to existing Test Methods D 3701, D 4808, and D 5291 for determining the hydrogen content of petroleum middle distillate products. It uses a low-resolution pulsed nuclear magnetic resonance (NMR) spectrometer. The boiling range of the distillates covered by the test method is 150 to 390°C. While the test method may be applicable to middle distillates outside this boiling range, in such cases the precision given in the method may not apply.

The test method is generally based on Test Methods D 3701 and D 4808, with a major difference being the use of a pulsed NMR spectrometer instead of a continuous wave NMR spectrometer.

TEST SUMMARY

A test specimen is analyzed in a pulsed NMR spectrometer calibrated with standard reference materials. The analyzer records in a nondestructive fashion the total NMR signal, which arises from the absolute amount of hydrogen atoms in the reference standards and the test sample. The absolute signal intensity is normalized by the corresponding sample mass. The resulting signal-per-gram ratios are used as a means of comparing theoretical hydrogen contents of the standards with that of the sample. To ensure an accurate measure of the absolute hydrogen content of the reference standards and sample, it is necessary to ensure that the measured hydrogen signal intensity is always directly proportional to the absolute hydrogen content of the standards and sample. Undercounting of the reference standard with respect to the sample is avoided by a number of strategies, including accurate filling into the linear response region of the sample compartment so that the mass recorded for the sample represents the true amount measured by NMR, and use of a recycle delay considerably greater than the longest relaxation time constant for the sample.

TEST PRECISION

The precision of this test method was determined at two temperatures: 35 and 40°C.

Temperature, °C	Repeatability	Reproducibility
35	$0.009352(X+1.7000)$	$0.1769(X+1.7000)$
40	$0.006409(X+5.0000)$	$0.01580(X+5.0000)$

Where X is the mass % hydrogen content.

Since there are no accepted reference materials for determining the bias of this test method, no statement on bias is being made.

HYDROGEN CONTENT OF PETROLEUM FRACTIONS: D 1018

This test method is applicable to petroleum fractions that can be burned completely without smoking in a wick lamp.

TEST SUMMARY

A sample is burned from a cotton wick in an atmosphere of purified air. The water formed is collected from the combustion gases by a desiccant and weighed.

TEST PRECISION

In the hydrogen content range of 11 to 16 mass %, this test method has a repeatability of 0.11 and a reproducibility of 0.18.

The bias of this test method has not be determined.

HYDROLYTIC STABILITY OF HYDRAULIC FLUIDS: D 2619

EXPLANATION

Hydrolytically unstable hydraulic fluids form acidic and insoluble contaminants which can cause hydraulic system malfunctions due to corrosion, valve sticking, or change in the viscosity of the fluid. This test method differentiates the relative stability of hydraulic fluids in the presence of water under the conditions of the test. The degree of correlation between this test and service performance has not been fully determined. This test method is applicable to petroleum or synthetic-base hydraulic fluids. Water-base or water-emulsion fluids can be evaluated by this test method, but additional water is not added to the sample.

TEST SUMMARY

Seventy-five g of sample is mixed with 25 g of water and along with a copper test specimen, are sealed in a pressure type beverage bottle. The bottle is rotated, end for end, for 48 h in an oven at 93°C(200°F). Layers are separated and insolubles are weighed. Weight change in copper is measured. Viscosity and acid number changes of fluid and acidity of water layer are determined.

TEST PRECISION

Property	Repeatability	Reproducibility
Copper Corrosion, mg/cm^3	0.3X	0.9X
Acid Number Change, mg KOH/g oil	0.8X	1.9X
Total Acidity of Water Layer, KOH/g oil	0.8X	1.3X

Where X is the mean value.

The bias of this test method is not available.

HYDROGEN SULFIDE IN LIQUEFIED PETROLEUM GAS (LPG) BY LEAD ACETATE METHOD:

D 2420

(Equivalent Test Methods: IP 401, ISO 8819, DIN 51855 T3, and AFNOR M41-011)

EXPLANATION

Liquefied petroleum gas and its combustion products must not be unduly corrosive to the materials that they come in contact with. Hence, the measurement of hydrogen sulfide in LPG is important. Also, in some specifications, for example, Specification D 1835, the odor of the gases must not be objectional. The sensitivity of the test is about 4 mg/m^3 of gas. Methyl mercaptan, if present, produces a transitory yellow stain on the lead acetate paper which, however, fades completely in less than 5 min. Other sulfur compounds present in LPG do not interfere.

TEST SUMMARY

The vaporized gas is passed over moist lead acetate paper under controlled conditions. Hydrogen sulfide reacts with lead acetate to form lead sulfide resulting in a stain on the paper varying in color from yellow to black, depending on the amount of hydrogen sulfide present.

TEST PRECISION

This is a pass-fail test and no statement regarding precision can be made.

This test method has no bias.

HYDROGEN SULFIDE IN RESIDUAL FUELS:

D 6021

EXPLANATION

Excessive levels of hydrogen sulfide in the vapor phase above residual fuel oils in storage tanks may result in health hazard, violation of OSHA limits, and public complaint. Also, it can cause corrosion in refining and other production activities. A concentration of 0.1 ppmw of this gas in the liquid phase of a residual fuel oil can generate about 10 to 100 ppmv in the vapor phase. Hence, an accurate method for low level determination in fuel oils is necessary.

A complementary method, Test Method D 5705, is a simple and consistent field test method for rapid determination of hydrogen sulfide in the vapor phase. However, it does not truly simulate the vapor phase concentration of a fuel storage tank. The current test gives more quantitative indication of potential exposure to hydrogen sulfide.

TEST SUMMARY

A residual fuel oil sample is placed in a headspace vial and heated in an oven at 60°C for more than 5 but less than 15 min. The headspace gas is sampled and injected into an apparatus capable of measuring hydrogen sulfide in the gaseous sample. There are two types of these apparatus, one using lead acetate (Test Methods D 4084 and D 4323), and the other based on chemiluminescence (Test Method D 5504). The procedure of heating and extracting the gas is repeated twice more.

TEST PRECISION

No data are available at present since the round robins have not been completed.

HYDROGEN SULFIDE IN RESIDUAL FUEL OILS: D 5705

EXPLANATION

See the discussion under Test Method D 6021. This test method covers the field determination of hydrogen sulfide in the vapor phase of a residual fuel oil sample. It is applicable to liquids with a viscosity range of 5.5 cSt at 40°C to 50 cSt at 100°C. The applicable range of hydrogen sulfide concentration is from 5 to 4000 ppm v/v.

TEST SUMMARY

A 1-L glass test bottle is filled to 50 volume % with fuel oil just prior to testing. In the test container, the vapor space above the fuel oil sample is purged with nitrogen to displace air. The test container with sample is heated in an oven at 60°C, and agitated on an orbital shaker at 220 r/min for 3 min. A length-of-stain detector tube and hand-operated pump are used to measure hydrogen sulfide concentration in the vapor phase of the test container.

Typically sulfur dioxide and mercaptans may cause positive interferences. In some cases, nitrogen dioxide can cause a negative interference. Most detector tubes will have a precleanse layer designed to remove certain interferences up to some maximum interferant level.

TEST PRECISION

Repeatability:	$0.20X+10$
Reproducibility:	$0.30X+15$

Where X is the average of the two results.

Bias of this test method has not been determined.

HYDROPEROXIDE NUMBER OF AVIATION TURBINE FUELS: D 6447

EXPLANATION

The magnitude of the hydroperoxide number is an indication of the quantity of oxidizing constituents present. Deterioration of fuel results in the formation of hydroperoxides and other oxygen-carrying compounds. The hydroperoxide number measures those compounds that will oxidize potassium iodide. The determination of hydroperoxide number is significant because of the adverse effect of hydroperoxides upon certain elastomers in the fuel systems.

This test method and Test Method D 3703 measure the same peroxide species, primarily the hydroperoxides in aviation fuels. The difference is that this test method does not use the ozone depleting substance 1,1,2-trichloro-1,2,2-trifluoroethane used in Test Method D 3703. This test method may also be applicable to any water-insoluble, organic fluid, particularly diesel fuels, gasolines, and kerosenes.

TEST SUMMARY

A quantity of sample is contacted with aqueous potassium iodide (KI) solution in the presence of acid. The hydroperoxides present are reduced by KI liberating an equivalent amount of iodine, which is quantified by voltammetric analysis.

TEST PRECISION

Repeatability:	$0.2153(X+0.0004)^{0.3997}$ mmole/L
Reproducibility:	$0.8036(X+0.0004)^{0.3997}$ mmole/L

Where X is the mean value.

No bias statement is being made due to the lack of a suitable reference material.

HYDROXYL NUMBER:
D 1957

EXPLANATION

Hydroxyl value is important in establishing reactivity with acids and isocyanates. It is also a measure of the degree of dehydration of castor oil.

This test method determines the total amount of residual hydroxyl groups present in oils and other fatty acid-containing materials, reported as hydroxyl value.

This test method involves the acetylation of hydroxyl-containing fatty oils and acids using pyridine as solvent. Other groups that will react with acetic anhydride such as primary and secondary amines under the conditions of the method will be reported as hydroxyl. Hydrocarbons, acetals, esters and water do not interfere. However, mercaptans, amines, some esters, and aldehydes interfere. Tertiary alcohols are incompletely determined. The hydroxyl value is expressed as milligrams of potassium hydroxide equivalent to the hydroxyl content of 1 g of the oil. A correction is applied for acid groups present.

This test method can be used for the determination of hydroxyl content of fatty oils, fatty alcohols, mono- and di-glycerides, and hydroxystearic acid.

TEST SUMMARY

A sample is acetylated with pyridine-acetic anhydride solution by refluxing for 1 h. Later neutralized *n*-butyl alcohol is added and the solution is titrated with alcoholic KOH solution to phenolphthalein end point.

TEST PRECISION

Repeatability:	2.4
Reproducibility:	3.0

INDIVIDUAL COMPONENTS IN ENGINE FUELS BY HIGH RESOLUTION GAS CHROMATOGRAPHY:
D 6729

EXPLANATION

Knowledge of the specified individual component composition (speciation) of gasoline fuels and blending stocks is useful for refinery quality control and product specification. Compliance in these areas may be determined through the use of this test method. This test method covers the determination of individual hydrocarbon components of spark-ignition engine fuels and their mixtures containing oxygenate blends (MTBE, ETBE, ethanol, etc.) with boiling ranges up to 225°C. Other light liquid hydrocarbon mixtures typically encountered in petroleum refining operations, such as blending stocks (napthas, reformats, alkylates, etc.) may also be analyzed. Individual component concentrations and precisions are determined in the range 0.01 to approximately 30 mass %. The test method also determines methanol, ethanol, t-butanol, MTBE, ETBE, TAME in engine fuels in the concentration range of 1 to 30 mass %.

TEST SUMMARY

Representative samples of the petroleum liquid are introduced into a gas chromatograph equipped with an open tubular (capillary) column coated with the specified stationary phase. Helium carrier gas transports the vaporized sample through the column, in which it is partitioned into individual components which are detected with a flame ionization detector as they elute from the end of the column. The detector signal is recorded digitally with an integrator or integrating computer. Each eluting component is identified by comparing its retention time to that established by analyzing reference standards or samples under identical conditions. The concentration of each component in mass % is determined by normalization of the peak areas after correction of selected components with detector response factors. The unknown components are reported individually and as a summary total.

TEST PRECISION

The repeatability and reproducibility estimates of individual components determination by this method are given in a format somewhat different than usually found in ASTM methods. See Annex A1.2 of Test Method D 6729 for this information.

The bias of this test method cannot be determined since an appropriate standard reference material for such analysis is not available.

INDIVIDUAL COMPONENTS IN ENGINE FUELS BY HIGH RESOLUTION GAS CHROMATOGRAPHY: **D 6730**

EXPLANATION

See Explanation in the Test Method D 6730 on the previous page.

TEST SUMMARY

A representative sample of the petroleum liquid is introduced into a gas chromatograph equipped with an open tubular (capillary) 100 meter column coated with a methyl silicone liquid phase modified with a capillary precolumn. Helium carrier gas transports the vaporized sample through the column, in which it is partitioned into individual components which are sensed with a flame ionization detector as they elute from the end of the column. The detector signal is presented on a strip chart recorder or digitally, or both, by way of an integrator or integrating computer. Each eluting component is identified by comparing its retention time to that established by analyzing reference standards or samples under identical conditions. The concentration of each component in mass % is determined by normalization of the peak areas after correction with detector response factors. Unknown components are reported as a total unknown mass %.

TEST PRECISION

Repeatability and Reproducibility of DHA Determinations.

NOTE 1—The following is a partial list of precision data that has been prepared by statisticians of CS94 in accordance with RR: D2-1007, and represents their best estimate of the cooperative study data. The complete precision dataset appears in Annex A1., Table A1.3.

NOTE 2—For each analyte to qualify for a precision statement, it must be present in at least six samples, and detected by at least six laboratories, at least once. The (repeatability standard deviation)/mean value for each analyte/sample combination must be less than or equal to 0.1, as per LOQ requirements which, while not a standard, is what CS94 is recommending.

NOTE 3—

Legend:

r_{min} = lower 95 % confidence limit of r_{est},
r_{est} = repeatability estimate in percent of concentration,
r_{max} = upper 95 % confidence limit of r_{est},
R_{min}, R_{est}, R_{max} = for reproducibility,
C_{min} = lower concentration limit that r_{est}, R_{est} is applicable, and
C_{max} = upper concentration limit that r_{est}, R_{est} is applicable.

Component	Average RI	r_{min}	r_{est}	r_{max}	R_{min}	R_{est}	R_{max}	C_{min}	C_{max}
n-butane	400.00	6.8	9.9	13.9	15.3	32.4	59.1	1.02	3.75
i-pentane	477.45	5.9	7.2	8.7	8.5	14.8	23.8	2.48	13.38
Pentene-1	490.83	5.2	7.5	10.5	9.7	13.8	19	0.06	0.43
n-pentane	500.00	5.2	6.5	8.1	7.1	10.4	14.8	1.06	3.49
Cyclopentane	566.84	3.8	4.9	6.2	7	10.1	14	0.07	0.59
2,3-dimethylbutane	569.24	2.9	3.2	3.5	5.1	8.5	13.1	0.7	1.91
n-hexane	600.00	2	2.4	2.9	3.6	5.1	6.9	0.33	2.52
Methylcyclopentane	625.86	2.2	2.6	3.1	4.5	6.4	8.7	0.37	2.35
1-methylcyclopentene	648.71	1.9	2.7	3.7	7.9	8.7	9.6	0.17	0.82
Benzene	649.92	2.6	3.6	4.8	5.5	9	13.7	0.17	1.58
Cyclohexane	657.81	2.7	3.7	4.9	8.2	14.8	24.3	0.07	0.9
2-methylhexane	667.61	1.6	2.2	2.9	5.1	6.1	7.2	0.39	1.09
2,2,4-trimethylpentane	688.48	2.4	3.2	4.1	7.4	11.4	16.7	0.1	11.26
n-heptane	700.00	2.5	3.4	4.5	7.7	10.8	14.7	0.21	1.06
Methylcyclohexane	717.89	2.8	3.4	4	4.1	5.9	8.2	0.11	1.2
2,3,4-trimethylpentane	746.83	2.3	3.8	6	5.8	7.8	10.3	0.08	4.26
Toluene	751.77	1.9	2.7	3.8	10.8	13.5	16.5	1.99	10.34
2-methylheptane	764.14	3.5	4.9	6.6	4.8	6.1	7.5	0.15	0.63
n-octane	800.00	2.2	3.6	5.5	6.5	15.7	30.9	0.14	0.75
Ethylbenzene	854.65	2.2	3.2	4.4	7.2	10.6	14.9	0.62	2.62
1,3-dimethylbenzene	864.22	2.6	3.3	4.2	9.7	12.5	15.7	1.55	6.66
3-methyloctane	880.24	5.1	8.5	13	8.7	15.5	24.9	0.07	0.29
n-nonane	900.20	3.9	6.4	9.8	8.6	10.3	12.2	0.06	0.34
n-propylbenzene	946.33	2.8	5	8.1	7.6	11.9	17.7	0.21	0.77
1,4-methylethylbenzene	956.22	3.5	5.3	7.7	5.1	7.7	11.1	0.32	1.19
1,3,5-trimethylbenzene	961.92	3.7	5.5	7.7	5.4	8.3	12.1	0.39	1.21
2-methylnonane	971.77	6.5	10.6	16.2	17.5	25.9	36.6	0.03	0.19
1,2,4-trimethylbenzene	983.40	4.2	5.7	7.5	7.8	10.6	13.9	1.19	4.32
n-decane	1000.20	7.5	9.2	11.1	12.1	17.9	25.3	0.03	0.25
1,2,3-trimethylbenzene	1006.88	3.8	5.8	8.5	7.2	8.5	10	0.28	0.96
n-undecane	1100.00	8.6	13.9	21	24.4	40	61.2	0.03	0.18
1,2,3,5-tetramethylbenzene	1108.79	6.4	7.8	9.3	10.2	13.9	18.3	0.21	0.51
Naphthalene	1168.01	6.1	8.5	11.3	12.9	16.9	21.5	0.13	0.4
n-dodecane	1200.00	12.2	16.7	22.1	20.2	32.9	50	0.01	0.11
2-methylnaphthalene	1282.57	7.6	11.1	15.4	17.5	22.3	28	0.05	0.5

INDIVIDUAL COMPONENTS IN ENGINE FUELS BY HIGH RESOLUTION GAS CHROMATOGRAPHY:

D 6733

EXPLANATION

This is a method similar to D 6729 and D 6730, except in this case a 50 meter capillary column is used. See further explanation under D 6729 on previous pages.

TEST PRECISION

Repeatability and Reproducibility for Individual Components.

	Range of Carbon	Range, % (m/m)	Repeatability, X (% (m/m))	Reproducibility, X (% (m/m))
Light Components	C4–C5	0.1–14	$0.04 \cdot X$	$0.16 \cdot X$
Paraffins	C6–C12	0.1–11.5	$0.01+0.03 \cdot X$	$0.04+0.07 \cdot X$
Naphthenes	C6–C8	0.1–3		
Olefins	C6–C8	0.1–1		
Aromatics	C6–C12	0.1–14	$0.05+0.02 \cdot X$	$0.1+0.06 \cdot X$

INSOLUBLES IN HYDRAULIC FLUIDS

D 4898

EXPLANATION

This test method indicates and measures the amount of insoluble contamination of hydraulic fluids. Minimizing the levels of insoluble contamination of hydraulic fluids is essential for the satisfactory performance and long life of the equipment. Insoluble contamination can not only plug the filters but can damage functional system components resulting in wear and eventual system failure. This method measures the insoluble contamination using a gravimetric method. The contamination determined includes both particulate and gel like matter, organic and inorganic, which is retained on a membrane filter disk of pore diameter as required by applicable specifications (usually 0.45 μm or 0.80 μm).

To indicate the nature and distribution of the particulate contamination, the gravimetric method should be supplemented by occasional particle counts of typical samples in accordance with Test Method F 312.

TEST SUMMARY

A given quantity of a fluid sample is passed through a membrane filter disk and the resultant weight increase of the filter is measured. The fluid sample is drawn through the filter by a vacuum and the insoluble contamination is collected on the surface of the filter. Additionally, the filter disk is microscopically scanned for excessively large particles, fibers, or other unusual conditions.

TEST PRECISION

Repeatability:	3.6 % of the weight of contamination per 100 mL of sample.
Reproducibility:	Not known
Bias	Cannot be determined

PENTANE INSOLUBLES BY MEMBRANE FILTRATION: D 4055

EXPLANATION

Pentane insolubles above 0.8 μm in size may lead to increased wear that can lead to premature equipment failure in critical applications. This test method covers the determination of pentane insolubles for particles exceeding 0.8 μm in new and used lubricating oils. Particles sizes above or below this limit can also be studied with appropriate size membrane filters; however, precision for such sizes is not known.

TEST SUMMARY

A sample of oil is mixed with pentane in a volumetric flask, and the oil solution is filtered through an 0.8 μm membrane filter. The flask, funnel, and the filter are washed with pentane to effect a complete transfer of particulates onto the filter. The filter with particulates is dried and weighed to give the pentane insolubles.

TEST PRECISION

Repeatability:	$0.177 \times X^{0.66}$
Reproducibility:	$0.759 \times X^{0.66}$

Where X is the average of two results.

The reproducibility of this test is very poor, and this test method is unsuitable for comparison of interlaboratory results.

The bias of this test method has not been determined.

INSOLUBLES IN USED LUBRICATING OILS: D 893

EXPLANATION

This test method covers the determination of pentane and toluene insolubles in used lubricating oils. Pentane insolubles can include oil-insoluble materials and some oil-insoluble resinous matter originating from oil or additive degradation, or both. Toluene insolubles can come from external contamination, fuel carbon, and highly carbonized materials from degradation of fuel, oil, and additives, or engine wear and corrosion materials. A significant change in pentane or toluene insolubles and insoluble resins indicates a change in oil which could lead to lubrication statem problems. The insolubles measured can also assist in evaluating the performance characteristics of a used oil or in determining the cause of equipment failure.

TEST SUMMARY

There are two test methods used. Procedure A covers the determination of insolubles without the use of coagulant in the pentane. It provides an indication of the materials that can be readily separated from the oil-solvent mixture by centrifugation. Procedure B covers the determination of insolubles in oils containing detergents, and employs a coagulant. In addition to the materials separated by using Procedure A, this coagulation procedure separates some finely divided materials that may be suspended in the oil.

The results obtained by Procedures A and B should not be compared since they usually give different values. The same procedure should be applied when comparing results obtained periodically on an oil in use, or when comparing results determined in different laboratories.

In Procedure A, a sample is mixed with pentane and centrifuged. The oil solution is decanted, and the precipitate washed twice with pentane, dried, and weighed. For toluene insolubles a separate sample of the oil is mixed with pentane and centrifuged. The precipitate is washed twice with pentane, once with toluene-alcohol solution, and once with toluene. The insoluble material is then dried and weighed.

In Procedure B, Procedure A is followed except that instead of pentane, a pentane-coagulant solution is used.

TEST PRECISION

Method	Range	Repeatability	Reproducibility
A-Pentane	0.0–1.0 %	0.07 %	0.10 %
A-Pentane	>1.0	10 % of Mean	15 % of Mean
A-Toluene		$0.216X^{0.5}$	$0.441X^{0.5}$
B-Toluene coagulated		$0.216X^{0.5}$	$0.957X^{0.5}$

Where X is toluene insolubles or coagulated toluene insolubles.

The reproducibility of this test is so poor that Procedure B coagulated toluene insolubles is unsuitable for comparing interlaboratory results.

This test method has no bias.

INSOLUBLES IN USED LUBRICATING OILS BY PAPER FILTERATION (LMOA METHOD):
D 7317

EXPLANATION

Coagulated pentane insolubles can include oil-insoluble materials, some oil-insoluble resinous matter originating from oil or additive degradation, soot from incomplete diesel fuel combustion, or a combination of all three. A significant change in the amount of coagulated pentane insolubles indicates a change in oil, and this could lead to lubrication system problems. Coagulated pentane insolubles measurements can also assist in evaluating the performance characteristics of a used oil or in determining the cause of equipment failure.

This test method was originally developed by Locomotive Maintenance Officers' Association (LMOA). This test method, in general, does not correlate with Test Method D 893 on insolubles in lubricating oils, since it uses separation by centrifugation and a more concentrated solution of anti-coagulant. The correlation between this test method and enhanced thermal gravimetric analysis procedure in Test Method D 5967 has not been investigated.

TEST SUMMARY

A sample of used lubricating oil is mixed with pentane–coagulant solution and filtered under vacuum. The filter is washed with pentane, dried, and weighed to give coagulated pentane insolubles.

TEST PRECISION

Repeatability = $0.061\ (X+1.77)$
Reproducibility = $0.237\ (X+1.77)$

where X is the average of two results.

The procedure ion this test method has no bias because results can only be defined in terms of this test method.

IODINE NUMBER:
D 2078

EXPLANATION

Iodine value is a measure of the unsaturation of the alkyl group or groups expressed in terms of percent iodine absorbed. This test method measures the unsaturation as iodine value in fatty quaternary ammonium chlorides by addition of an iodine/chlorine reagent. The amount of reagent absorbed is determined by back titrating the excess reagent and comparing it to a blank determination. This determination is an indication of the source of the fatty component or, if the source is known, the number of the fatty components (for example, 1, 2, 3, or 4), in the quaternary ammonium chloride. This test method is essentially equivalent to Test Methods D 1959 and D 2075.

TEST SUMMARY

A sample is dissolved in chloroform and reacted with Wijs solution for 30 min in the dark at 25°C. At the end, the solution is mixed with potassium iodide (KI) and titrated with sodium thiosulfate using starch indicator.

TEST PRECISION

The precision or bias data have not been obtained; however, this test method has been in use for many years, and its usefulness has been well established.

IRON CHIP CORROSION FOR WATER DILUTABLE METAL WORKING FLUIDS:
D 4627

EXPLANATION

This test method evaluates the ferrous corrosion control characteristics of water-dilutable metalworking fluids. The results obtained by this test method are a useful guideline in determining the ability of water-diluted metalworking fluids to prevent or minimize rust under specific conditions. There is usually a relationship between the results of this test and a similar ability of the subject coolant to prevent rust on nested parts or in drilled holes containing chips, etc. However, the conditions, metal types, etc., found in practice will not correlate quantitatively with these controlled laboratory conditions. The procedure may not be able to differentiate between two products with poor rust control due to the wide spacing between test dilutions.

TEST SUMMARY

Cast iron chips are placed in a petri dish containing a filter paper and diluted metalworking fluid. The dish is covered and allowed to stand overnight. The amount of rust stain on the filter paper is an indication of the corrosion control provided by the fluid.

TEST PRECISION

Repeatability	0.64
Reproducibility	1.85

These data are for sample Nos. 1, 3, 4, and 5.

This test method has no bias because the corrosion value can be defined only in terms of this test method.

LEAD DETERMINATION IN GASOLINE

GENERAL

With the phaseout of lead in gasoline in the U.S.A. over the last decade, the importance of determination of added lead in gasolines has decreased. However, it is still necessary to measure the compliance of trace lead as required by federal regulations for lead-free gasoline. The methods available for this work include use of atomic absorption, X-ray fluorescence, and wet chemistry methods.

LEAD IN GASOLINE BY ATOMIC ABSORPTION SPECTROMETRY (AAS):
D 3237
(Equivalent Test Method: IP 428)

This test method is applicable in the range 2.5 to 25 mg lead/L. This test method compensates for variations in gasoline composition and is independent of lead alkyl type.

TEST SUMMARY

The gasoline sample is diluted with methyl isobutyl ketone and the alkyl lead components are stabilized by reaction with iodine and a quaternary ammonium salt. The lead content of the sample is determined by flame atomic absorption spectrometry at 283.3 nm, using the standards prepared from reagent grade lead chloride. All alkyl lead compounds give identical response.

TEST PRECISION

This test method has a repeatability of 1.3 mg/L and a reproducibility of 2.6 mg/L.

This test method has been shown to have no bias.

LEAD DETERMINATION IN GASOLINE BY IODINE CHLORIDE (ICl) METHOD:

D 3341

(Equivalent Test Methods: IP 270, ISO 3830, DIN 51769 T2, and JIS K 2255)

This test method is applicable in the range 0.026 to 1.3 g Pb/L.

TEST SUMMARY

A known volume of the sample is diluted with heavy distillate, and shaken with aqueous iodine monochloride reagent. Thus, any tetraalkyl lead compounds present convert to dialkyl lead compounds and are extracted into the aqueous phase. The separated aqueous extract is evaporated to low volume to decompose free iodine mono-chloride. Any organic matter present is removed by oxidation with nitric acid, which also converts dialkyl lead compounds to inorganic lead compounds. The residue is dissolved in water and buffered at pH 5. The lead content of this solution is determined by titrating with EDTA using xylenol orange as an indicator.

TEST PRECISION

This test method has a repeatability of $0.00365+0.0073X$ and a reproducibility of $0.0135+0.027X$ where X is the average result in g lead/L at 15.5°C.

The bias of this test method has not been determined due to lack of cooperators.

LEAD DETERMINATION IN GASOLINE BY X-RAY FLUORESCENCE (XRS):

D 5059

These test methods cover the determination of lead in gasoline in the range of 0.0026 to 1.32 g lead/L. These test methods compensate for the normal variation in gasoline composition and are independent of lead alkyl type. Three parts of this test method had different designations earlier. Test Method A was formerly Methods D 2599 Sections 5–9; Test Method B was D 2599 Sections 10–14; and Test Method C was D 3229 Sections 15–19.

TEST SUMMARY

Test Method A (*Bismuth Internal Standard Method for High Concentration*)—One volume of the sample is mixed with an equal volume of bismuth internal standard solution. The mixture is placed in an X-ray beam and radiations at 1.75 A for lead and 1.144 A for bismuth are measured. By comparing the ratio of intensities to that of known standards, the lead content of gasoline is calculated.

Test Method B (*Scattered Tungsten Radiation Method*)—The ratio of the net X-ray intensity of lead radiation to the net intensity of the incoherently scattered tungsten radiation is obtained on a portion of the sample. By comparing this to the intensity of a known lead standard, the lead content of gasoline is calculated.

Test Method C (*Bismuth Internal Standard Method for Low Concentration*)—This test method is the same as in Test Method A, but additionally a background radiation at 1.194 Å is also measured. A blank made with isooctane and bismuth internal standard is run using the same procedure. The lead content of gasoline is calculated based on comparing the ratio of lead to bismuth radiation intensities to that of known standards, after subtracting the blank ratio.

TEST PRECISION

Test Method	Repeatability	Reproducibility
A	$0.008+0.008X$	$0.27+0.30X$
B	$0.004+0.015X$	$0.010+0.039X$
C	$0.007+0.14X$	$0.018+0.15X$

Where X is the average result expressed in g lead/L at 15°C in Test Methods A and B, and in g lead/US gallon at 15.5°C in Test Method C.

These test methods have no bias.

LEAD DETERMINATION RAPID FIELD TEST FOR TRACE LEAD IN GASOLINE:
D 3348

This test method is based on the use of Mobil lead test kit and is intended for field use by nontechnical personnel, for the quantitative measurement of lead in unleaded gasoline in the range from 2.64 to 26.4 mg Pb/L. This test method is applicable to all commercial gasolines and responds to all types of lead alkyls as well as other organic and inorganic forms of lead. This is a screening test and is not intended to replace other lead measurement methods.

TEST SUMMARY

The gasoline sample is treated with iodine and tetraethyl ammonium chloride in chloroform and subjected to ultraviolet light. The lead alkyls form water soluble lead alkyl iodides, which are extracted out by shaking with an aqueous ammonium nitrate solution. The aqueous extract is filtered into a solution of 4-(2-pyridylazo)-resorcinol di-sodium salt (PAR) and ammonium hydroxide. The lead-PAR complex is measured colorimetrically at 490 nm using a previously prepared calibration curve. PAR reagent also reacts with many other transition metals forming colored complexes; however, none of these metals are normally present in a soluble form in gasoline.

TEST PRECISION

This test method has a repeatability of 2.64 mg/L and a reproducibility of 5.28 mg/L.

LEAKAGE TENDENCIES OF AUTOMOTIVE GREASES:
D 1263

EXPLANATION

This test method evaluates the leakage tendencies of wheel bearing greases. This test method provides a screening device that permits differentiation between products having distinctly different leakage characteristics. It is not equivalent to long term service tests, nor is it intended to distinguish between wheel bearing greases showing similar or borderline leakage.

TEST SUMMARY

The grease is distributed in a modified front-wheel hub and spindle assembly. The hub is rotated at a speed of 660 ± 30 r/min for 6 h ± 5 min, at a spindle temperature, which is raised to and then maintained at 105 ± 1.2°C. Leakage of grease or oil, or both, is measured, and the condition of the bearing surface is noted at the end of the test.

TEST PRECISION

Leakage in the area of	2 g	15 to 20 g
Repeatability:	1.5 g	9 g
Reproducibility:	4 g	9 g

This test method has no bias.

LEAKAGE TENDENCIES OF GREASES:
D 4290

EXPLANATION

This test method differentiates among wheel bearing greases having distinctly different high temperature leakage characteristics. It is not the equivalent of longtime service tests. This test method has proven to be helpful in screening greases with respect to leakage tendencies for automotive wheel bearing applications. It is possible for skilled operators to observe changes in grease characteristics that can occur during the test, such as grease condition. Leakage is reported as a quantitative value, whereas the evaluation grease condition is subject to differences in personal judgment among operators and cannot be used effectively for quantitative measurements.

TEST SUMMARY

The test grease is distributed in a modified, automobile front wheel hub-spindle-bearings assembly. While the bearings are thrust-loaded to 111 N, the hub is rotated at 1000 r/min and the spindle temperature is maintained at 160°C for 20 h. Leakage of grease or oil, or both, is measured, and the condition of the bearing surface is noted at the end of the test.

TEST PRECISION

Repeatability	1.505($X^{0.5}$)
Reproducibility	3.848($X^{0.5}$)

Where X is the average of the two test results.

This test method has no bias because the value of grease leakage can be defined only in terms of this test method. In the round robin to obtain precision, testers made by Pam and Koehler were used. Both testers gave equivalent results.

LIFE PERFORMANCE OF GREASES:
D 3527

EXPLANATION

This test method differentiates among wheel bearing greases having distinctly different high temperature characteristics. It is not the equivalent of long-time service tests, nor is it intended to distinguish between the products having similar high-performance properties. This test method has proven to be helpful in screening greases with respect to life performance for automotive wheel bearing applications. Changes to this test method in the 1985 revision increased die test severity. Results will not be comparable with data from earlier procedures.

TEST SUMMARY

The test grease is distributed in the bearings of a modified, automobile front wheel hub-spindle-bearing assembly. While the bearings are thrust-loaded to 111 N, the hub is rotated at 1000 r/min and the spindle temperature maintained at 160°C for 20 h, 4 h off operating cycle. The test is terminated when grease deterioration causes the drive motor torque to exceed a calculated motor cut off value. Grease life is expressed as the accumulated on-cycle hours.

TEST PRECISION

Repeatability:	(0.8)X
Reproducibility:	(1.2)X

Where X is the average of two test results.

This procedure has no bias because the value of grease life can only be defined in terms of the test method. In the round robin to determine the precision, three types of testers were used: PAM, Koehler, and Falex. All testers gave equivalent results.

LINEAR FLAME PROPAGATION RATE OF LUBE OILS AND HYDRAULIC FLUIDS:
D 5306

EXPLANATION

The linear flame propagation rate of a sample is a property that is relevant to the overall assessment of flammability or relative ignitability of fire resistance lubricants and hydraulic fluids. It is intended to be used as a bench scale test for distinguishing between the relative resistance to ignition of such materials. It is not intended to be used for the evaluation of the relative flammability of flammable, extremely flammable, or volatile fuels, solvents, or chemicals.

This test method covers the determination of the linear flame propagation rates of lubricating oils and hydraulic fluids supported on the surface of and impregnated into ceramic fiber media. The data thus generated are to be used for the comparison of relative flammability. This test method should be used to measure and describe the properties of materials, products, and assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions, However, results of this test method may be used as elements of fire risk which takes into account all of the factors that are pertinent to an assessment of the fire hazard of a particular end use.

TEST SUMMARY

A section of a ceramic fiber support medium (string) is impregnated with the sample under specific conditions. The impregnated fiber is placed on a standard support. The sample is ignited and the time required for the flame front to propagate across a measured distance is determined by means of a thermoelectric system. The average propagation rate is then calculated from the measured distance of the flame travel and the time required for the flame front to propagate over that distance.

TEST PRECISION

The precision of this test method based on use of different kinds of fluids is given in Table A1.1 of the Test Method D 5306. Since it is a complex table, it is not reproduced here.

No bias statement is possible because the linear flame propagation rate can be defined only in terms of this test method.

LITHIUM AND SODIUM IN GREASES BY FLAME PHOTOMETER:

D 3340

EXPLANATION

Lubricating greases can contain the lithium soap of hydroxy stearic acid or the sodium soap of various fatty acids as thickeners. The determination of total lithium or sodium is a measure of the amount of thickener in the grease. This test method uses flame photometry for this determination.

TEST SUMMARY

The sulfated ash (Test Method D 874) of the grease sample is extracted with water and the lithium and sodium content of the resulting solution is determined by means of a suitable flame photometer.

TEST PRECISION

Metal	Range, mass %	Repeatability	Reproducibility
Lithium	0 to 0.4	$1.014X^{2/5}$	$0.0175X^{2/5}$
Sodium	0 to 8	$0.029X^{2/5}$	$0.166X^{2/5}$

Where X is the average of determinations.

The bias of this test method has not been determined.

LOAD-CARRYING CAPACITY OF GREASES:

D 2509

EXPLANATION

This test method is widely used for specification purposes and is used to differentiate between greases having low, medium, or high levels of extreme pressure characteristics. The test results may not correlate with results from service. The test uses a Timken Extreme Pressure Tester.

TEST SUMMARY

The tester is operated with a steel test cup rotating against a steel test block. The rotating speed is 123.71 ± 0.77 m/min, which is equivalent to a spindle speed of 800 ± 5 r/min. The grease samples are brought to and applied at 24 ± 6°C (75 ± 10°F). Two determinations are made: the minimum load that will rupture the lubricant film being tested between the rotating cup and the stationary block and cause abrasion; and the maximum load at which the rotating cup will not rupture the lubricant film and cause abrasion between the rotating cup and the stationary block.

TEST PRECISION

Repeatability:	23 % of the mean
Reproducibility:	59 % of the mean

This test method has no bias.

LOW TEMPERATURE FLUIDITY AND APPEARANCE OF HYDRAULIC FLUIDS:
D 6351

EXPLANATION

The temperature at which a lubricant remains fluid and homogenous after seven days is an index of its ability to withstand prolonged exposure to cold temperatures. Quick cool, short term tests such as Test Methods D 97 and D 2500 do not adequately predict the tendency to solidify over long time spans at cold temperatures. This test method covers the fluidity and appearance of hydraulic fluids after storage at low temperatures. This test method, however, is not intended to indicate cold temperature pumpability performance. No specific temperature of measurement is given in this test method because fluids with different viscosity grades have different cold temperature performance expectations.

TEST SUMMARY

The sample is dried to remove trace amounts of water and is then cooled to a specified temperature. After seven consecutive days, the sample is examined for its ability to flow and observed for homogeneity.

TEST PRECISION

Precision data are not yet available.

No statement of bias can be made.

LUBRICATING GREASES ANALYSIS:
D 128

These test methods cover conventional greases that consist essentially of petroleum oil and soap. Thus these test methods are applicable to many but not all greases. Composition should not be considered as having any direct bearing on service performance unless such correlationship is established. The constituents covered by these test methods are soap, unsaponifiable matter (petroleum oil, etc.), water, free alkalinity, free fatty acid, fat, glycerin, and insolubles.

A supplementary test method is also provided intended primarily for application to greases containing thickeners that are essentially insoluble in *n*-hexane, and to greases that cannot be analyzed by conventional methods because of the presence of such constituents as non-petroleum fluids or non-soap type thickeners, or both.

No round robins have been completed to obtain precision and bias data.

LUBRICITY OF AVIATION TURBINE FUELS:
D 5001

EXPLANATION

Wear due to excessive friction resulting in shortened life of engine components such as fuel pumps and fuel controls have sometimes been ascribed to lack of lubricity in an aviation fuel. The relationship of test results to aviation fuel system component distress due to wear has been demonstrated for some fuel/hardware combinations where boundary lubrication is a factor in the operation of the component. The wear scar generated in the ball-on-cylinder lubricity evaluator (BOCLE) test is sensitive to contamination of the fluids and test materials, the presence of oxygen and water in the atmosphere, and the temperature of the test. Lubricity measurements are also sensitive to trace materials acquired during sampling and storage. The BOCLE test may not directly reflect operating conditions of engine hardware. Some fuels that contain a high content of certain sulfur compounds may give anomalous test results.

TEST SUMMARY

The test fluid is placed in a test reservoir in which air is maintained at 10 % relative humidity. A nonrotating steel ball is held in a vertically mounted chuck and forced against an axially mounted steel ring with an applied load. The test cylinder is rotated at a fixed speed while being partially immersed in the fluid reservoir. The wear scar generated on the test ball is the measure of fluid lubricating properties.

TEST PRECISION

Repeatability:	$0.109X^{1.8}$
Reproducibility:	$0.167X^{1.8}$

Where X is the wear scar diameter in mm.

This test method has no bias.

LUBRICITY OF DIESEL FUELS BY HIGH-FREQUENCY RECIPROCATING RIG (HFRR):

D 6079

EXPLANATION

Diesel fuel injection equipment has some reliance on lubricating properties of the diesel fuel. Shortened life of engine components such as diesel fuel injection pumps and injectors, has sometimes been ascribed to lack of lubricity in a diesel fuel. This test method evaluates the diesel fuel lubricity using a high frequency reciprocating rig (HFRR). It is not known whether this test will predict the performance of all additive/fuel combinations. Work is underway to establish this correlation. The wear scar generated in the HFRR test is sensitive to contamination of the fluids and test materials and the temperature of the test. Lubricity evaluations are also sensitive to trace contaminants acquired during test fuel sampling and storage. The HFRR test may be used to evaluate the relative effectiveness of diesel fuels for preventing wear under the prescribed test conditions.

TEST SUMMARY

A 2-mL test specimen of fuel is placed in the test reservoir of the HFRR and adjusted to either of the standard temperatures (25 or 60°C). The preferred test temperature is 60°C unless there is concern regarding the volatility or degradation of the fuel at that temperature. When the fuel temperature has stabilized, a vibrator arm holding a nonrotating steel ball and loaded with a 200-g mass is lowered until it contacts a test disk completely submerged in the fuel. The ball is caused to rub against the disk with a 1-mm stroke at a frequency of 50 Hz for 75 min. Then the ball is removed from the vibrator arm and cleaned. The dimensions of the major and minor axes of the wear scar are measured under 100× magnification and recorded.

TEST PRECISION

This test method has a repeatability of 0.062 mm at 25°C and 0.080 mm at 60°C, and a reproducibility of 0.127 mm at 25°C and 0.136 mm at 60°C.

This test method has no bias.

LUMINOMETER NUMBERS OF AVIATION TURBINE FUELS:

D 1740

EXPLANATION

This test method provides an indication of the relative radiation emitted by the combustion products of gas turbine fuels from a diffusion flame. The radiation level is expressed as luminometer number, which is related to the hydrocarbon type composition of such fuels. A high luminometer number indicates a fuel of low radiation characteristics. Because radiant heat transfer exerts a strong influence on the metal temperature of combustion liners and other hot section parts of gas turbines, the luminometer numbers provide a basis for the correlation of fuel characteristics with the life of these components. There is a good correlation between luminometer numbers by this test and smoke point (Test Method D 1322).

TEST SUMMARY

The luminometer number of a fuel is determined by burning the fuel in the ASTM-CRC Luminometer lamp and obtaining a curve of flame radiation, as measured by the optical filter and photo cell unit against the temperature rise across the burner measured by a thermocouple placed just above the flame. This temperature rise is compared with that obtained on a pair of reference fuels at a constant radiation level. Luminometer number is calculated from the difference in temperature rise between the test fuel and for tetralin divided by the difference in temperature rise for isooctane and tetralin.

TEST PRECISION

Repeatability:	6.1 luminometer numbers
Reproducibility:	8.8 luminometer numbers

This test method has no bias.

MANGANESE IN GASOLINE BY AAS: D 3831

EXPLANATION

Certain organo-manganese compounds act as anti-knock agents when added to gasoline, usually as methylcyclopentadienyl manganese tricarbonyl (MMT). This test method covers the determination of total manganese content of gasoline in the concentration range from 0.25 to 0.30 mg Mn/L. This test method is not applicable to gasoline containing highly cracked material (>20 bromine numbers).

TEST SUMMARY

The gasoline sample is treated with bromine and diluted with methyl isobutyl ketone. The manganese content of the solution is determined by atomic absorption spectrometry using an air-acetylene flame at 279.5 nm and standards prepared from an organo-manganese standard material.

TEST PRECISION

Repeatability:	$0.3308(X+0.1062)^{0.5}$
Reproducibility:	$1.650(X+0.1062)^{0.5}$

Where X is the mean of duplicate results.

The bias of this test method has not been determined.

MELTING POINT OF PETROLEUM WAX: D 87

(Equivalent Test Methods: IP 55, ISO 3841, and DIN 51570)

EXPLANATION

Melting point (cooling curve) is a test widely used by wax suppliers and consumers. It is particularly applied to petroleum waxes that are rather highly paraffinic or crystalline in nature. A plateau occurs with specimens containing appreciable amounts of hydrocarbons that crystallize at the same temperature, giving up heat of fusion, thus temporarily retarding the cooling rate. Generally, petroleum waxes with large amounts of nonnormal hydrocarbons or with amorphous solid forms will not show the plateau. This test method is unsuitable for the waxes of the petrolatum group, microcrystalline waxes, or blends of such waxes with paraffin wax or scale wax. Other methods used for petroleum waxes are Test Methods D 127 and D 938. However, results may differ depending on the test method used.

TEST SUMMARY

A molten wax specimen is placed in a test tube fitted with a thermometer and placed in an air bath, which in turn is surrounded by a water bath held at 16 to 28°C(60 to 80°F). As the molten wax cools, periodic readings of its temperature are taken. When solidification of the wax happens, the rate of temperature decreases, yielding a plateau in the cooling curve. The temperature at that point is recorded as the melting point (cooling curve) of the sample.

TEST PRECISION

Apparatus	Repeatability	Reproducibility
Manual	0.11°C	0.41°C
Automatic	0.23°C	0.50°F

This test method has no bias.

DROP MELTING POINT OF PETROLEUM WAX: D 127

(Equivalent Test Methods: IP 133 and ISO 6244)

EXPLANATION

Drop melting point test method is often used to measure the melting characteristics of petrolatums and other high viscosity petroleum waxes.

TEST SUMMARY

Specimens are deposited on two thermometer bulbs by dipping chilled thermometers into the sample. Then these thermometers are placed in test tubes and heated in a water bath until the specimens melt and the first drop falls from each thermometer bulb. The average of the temperatures at which these drops fall is the drop melting point of the sample.

TEST PRECISION

Repeatability:	0.8°C(1.4°F)
Reproducibility:	1.3°C(2.4°F)

This test method has no bias.

MERCAPTAN SULFUR IN PETROLEUM PRODUCTS: D 3227

(Equivalent Test Methods: IP 342, ISO 3012, JIS K 2276, and AFNOR M07-022)

EXPLANATION

Mercaptan sulfur has an objectionable odor, an adverse effect on fuel system elastomers, and is corrosive to fuel system components. This test method covers the determination of mercaptan sulfur in gasolines, kerosenes, aviation turbine fuels, and distillate fuels containing from 0.0003 to 0.01 mass % of mercaptan sulfur. Organic sulfur compounds such as sulfides, disulfides, and thiophene do not interfere. Elemental sulfur in amount <0.0005 mass % does not interfere. Hydrogen sulfide will interfere if not removed by precipitating with cadmium sulfate.

TEST SUMMARY

The hydrogen sulfide-free sample is dissolved in an alcoholic sodium acetate titration solvent and titrated potentiometrically with silver nitrate solution, using as an indicator the potential between a glass reference electrode and a silver/silver-sulfide indicating electrode. Under these conditions, the mercaptan sulfur is precipitated as silver mercaptide and the end point of the titration is shown by a large change in cell potential.

TEST PRECISION

Repeatability:	$0.00007+0.027X$
Reproducibility:	$0.00031+0.042X$

Where X is the average mercaptan sulfur, mass %.

The bias of this test method has not been determined.

MISTING PROPERTIES OF LUBRICATING FLUIDS: D 3705

EXPLANATION

This test provides a guide for evaluating the misting properties of oils used in industrial mist lubrication systems. The degree of correlation between this test and service performance has not been fully determined. This test method should not be used to evaluate fluids containing solid additives such as graphite.

TEST SUMMARY

The mist generator is charged with oil and installed in the mist system. The unit is operated for 19 h. The mist generator, line condensate bottles, and reclassified oil collector are weighed before and after the test. The output from the generator and percentages of reclassified oil, line condensate, and stray mist are determined from changes in weight.

TEST PRECISION

Results	Repeatability	Reproducibility
Oil Output, g/h	8 %	41 %
Reclassified Oil, %	6 %	57 %
Line Condensate, %	22 %	73 %

This test method has no bias.

METAL ANALYSIS BY SPECTROSCOPY:

GENERAL

In addition to the naturally occurring metals in the crude oil and the petroleum products derived from it, certain metals are added as additives to lubricating oils and similar products. These metal additives act as detergents, antioxidants, antiwear agents, etc. Some of these additives contain one or more of these metals: boron, barium, calcium, copper, magnesium, molybdenum, phosphorus, zinc, etc. In used lubricating oils, several other metals may be present coming from the engine wear in addition to those from the additives. On the other hand, in certain petroleum products even trace amounts of metals may prove deleterious to engine performance. Hence, it is important to have test methods that can determine metals, both at trace levels and at major concentrations. Elsewhere in this guide, some specific test methods are described for the determination of specific metals such as lithium, manganese, sodium, etc. In this particular section, multielement methods using atomic spectroscopic techniques are described. In the current marketplace atomic absorption spectrometry, inductively coupled plasma atomic emission spectrometry, and X-ray fluorescence spectroscopy have proved widely popular and highly useful.

TRACE METALS IN GAS TURBINE FUELS BY AA/FES: D 3605

(Equivalent Test Methods: IP 413, ISO 8691, and DIN 51790 T3)

EXPLANATION

Knowledge of the presence of trace metals in gas turbine fuels enables the user to predict performance and, when necessary, to take appropriate action to prevent corrosion. This test method covers the determination of sodium, lead, calcium, and vanadium in Specification D 2880, Grade Nos. 1-GT and 2-GT fuels in the range from 0.1 to 2.0 mg/L. This test method is intended for the determination of oil-soluble metals and not waterborne contaminants in oil-water mixtures.

TEST SUMMARY

To eliminate the problems encountered with the direct analysis of typical gas turbine fuels that exhibit significant variations in physical properties, the method of standard additions is used. Lead is determined by AAS and sodium by AAS or AES in an air-acetylene flame. Calcium and vanadium are determined by AAS or AES in a nitrous oxide-acetylene flame. If a user is interested in analyzing potassium, a procedure similar to that for sodium should be used.

Most experience with this test method is using AAS and the precision given is based on AAS measurements only.

TEST PRECISION

In the range 0.1 to 0.5 mg/L concentration, the following precision has been found for AAS measurements.

Element	Repeatability	Reproducibility
Vanadium	$0.452X^{0.5}$	$0.616X^{0.5}$
Lead	$0.244X^{0.5}$	$0.900X^{0.5}$
Calcium	$0.202X^{0.5}$	$0.402X^{0.5}$
Sodium	$0.232X^{0.5}$	$0.738X^{0.5}$

Where X is the average of duplicate results.

The bias for this method has not been determined.

METAL ANALYSIS IN LUBRICATING OILS BY AAS: **D 4628** (Equivalent Test Methods: IP 308 and DIN 51391 T1)

TEST SUMMARY

This test method is applicable for the determination of mass percent barium from 0.005 to 1.0 %, zinc, calcium, and magnesium from 0.002 to 0.3 % in lubricating oils. Higher concentrations can be determined by appropriate dilution. Levels at 100 ppm concentrations are also allowed to be analyzed.

A sample is weighed and base oil is added to 0.25 g total weight. Fifty mL of a kerosene solution, containing potassium as an ionization suppressant, are added, and the sample and oil are dissolved. Standards are similarly prepared, always adding oil if necessary to yield a total weight of 0.25 g. These solutions are burned in the flame of an atomic absorption spectrophotometer. An acetylene/nitrous oxide flame is used.

The presence of certain viscosity improvers (VI) may cause a negative bias for some metals. It is suggested to (*a*) add the same VI to both the sample and calibration standard, or more easily (*b*) dilute the VI containing the sample until the VI effect is no longer significant.

TEST PRECISION

Element	Range, Weight %	Repeatability	Reproducibility
Ba	0.005–1.0	$0.0478X^{0.66}$	$0.182X^{0.66}$
Ca	0.002–0.3	$0.0227X^{0.66}$	$0.0779X^{0.66}$
Mg	0.002–0.3	$0.0168X^{0.66}$	$0.0705X^{0.66}$
Zn	0.002–0.2	$0.0247X^{0.66}$	$0.0537X^{0.66}$
Ca	1.7	0.032	0.090
Zn	1.0	0.025	0.048

Where X is the average of duplicate results.

METAL ANALYSIS ALUMINUM AND SILICON IN FUEL OILS BY INDUCTIVELY COUPLED PLASMA (ICPAES) AND AAS:

D 5184

(Equivalent Test Methods: IP 377, ISO 10478, and DIN 51416)

EXPLANATION

Catalyst fines in fuel oils can cause abnormal engine wear. These test methods provide a means of determining silicon and aluminum, the major constituents of the catalysts. The concentration range determined is between 5 and 150 mg/kg for aluminum and 10 to 250 mg/kg for silicon.

TEST SUMMARY

Two test methods are available, one using ICPAES, and the other using flame AAS. A sample is heated in a platinum dish, the combustible material is removed by burning, and the carbon finally removed by heating in a muffle furnace at a temperature of 550±25°C. The residue is fused with a lithium tetraborate/lithium fluoride flux. The fused mixture is digested in a solution of tartaric and hydrochloric acids, and diluted to volume with water. The aluminum and silicon in the resultant solution are measured with either atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry.

TEST PRECISION

Element	Range, mg/kg	Repeatability, mg/kg		Reproducibility, mg/kg	
		ICPAES	AAS	ICPAES	AAS
Aluminum	7–139	$0.0660X$	$0.2968X^{2/3}$	$0.337X$	$0.789X^{2/3}$
Silicon	10–236	$0.0643X$	$0.3539X^{2/3}$	$0.332X$	$1.338X^{2/3}$

Where X is the mean concentration in mg/kg.

The bias of this test method has not been determined.

TRACE ELEMENTS IN MIDDLE DISTILLATE FUELS BY ICP-AES:

D 7111

EXPLANATION

Trace metals in turbine fuels can cause corrosion and deposition on turbine components at elevated temperatures. Some diesel fuels have specification limit requirements for trace metals to guard against engine deposits. For example, trace level copper in middle distillate aviation turbine fuel can significantly accelerate thermal instability of the fuel leading to oxidation and production of detrimental insoluble deposits in the engine. Gas turbine fuel oil Specification D 2880 provides recommended upper limits for five trace metals (calcium, lead, potassium, sodium, and vanadium). Military specification MIL-16884J for naval distillate fuel sets requirements for maximum concentrations of the same five metals. Both specifications designate Test Method D 3605, an atomic absorption/flame emission method for the quantitative determination of four of these metals. It does not include potassium in its scope. This test method offers an alternative to D 3605 and can also determine potassium as well as several other trace elements.

There are several sources of multi-element contamination of naval distillate fuel. Sea water is pumped into the diesel fuel tanks (as ballast) to trim ships. Also, some of the oilers (fuel supply ships) have dirty tanks. Corrosion products come from unlined tanks, piping, pumps, and heat exchangers.

This test method can determine about 20 trace elements in the range 0.1 to 2.0 mg/kg in middle distillate fuels using inductively coupled plasma-atomic emission spectrometry (ICP-AES). However, the test method can be used for concentrations about this range. The middle distillate fuels covered in this test method have all distillation fractions contained within the boiling range of 150 to 390°C. This includes, but is not limited to, diesel fuels and aviation turbine fuels. This test method is not intended to analyze insoluble particulates. However, very small particulate matter (smaller than 1μ) will be carried into the plasma and be included in the quantitative analysis. This test method may give a result that is higher than the true value if an analyte is present in the sample in a form which is sufficiently volatile. For example, hexamethylsiloxane will generate a biased high result for silicon.

TEST METHOD SUMMARY

Calibration standards are prepared by mixing organometallic standard materials in kerosene. An internal standard material is added to both the calibration standards and the fuel samples. The calibration standards and the fuel samples are aspirated into the ICP-AES instrument. The concentrations of the elements in the fuel are calculated by comparing emission intensity ratios of the fuel and the calibration standards to the internal standard.

INTERFERENCES—The elemental wavelengths listed in the test method have been found to be free of spectral interference. If a spectral interference does exist, then selecting an analytical wavelength other than the ones listed in the test method may be used as long as the new wavelength possesses appropriate sensitivity for the scope of the method. Alternatively, the ICP spectrometer manufacturer's software may be used to provide corrections to interferences that cannot be avoided by wavelength selection and background correction. An empirical method of correcting for spectral interferences is detailed in Test Method D 5185.

TEST PRECISION

Since there is no accepted reference materials for determining bias for this test method, no statement on bias is being made.

Repeatability.

Element	Range,[a] *mg/kg*	Repeatability,[b] *mg/kg*
Aluminum	0.13–1.77	$0.9692X^{0.5202}$
Barium	0.11–1.92	$0.05896X^{0.7520}$
Calcium	0.10–1.77	$0.0887X^{0.7391}$
Chromium	0.11–1.73	$0.05839X^{1.0350}$
Copper	0.11–1.85	$0.03470(X+0.6579)$
Iron	0.11–1.71	$0.05312X^{0.9114}$
Lithium	0.10–1.83	$0.06941(X-0.02484)$
Lead	0.08–1.73	$0.1166X^{0.6934}$
Magnesium	0.10–1.76	$0.04820X^{0.6570}$
Manganese	0.10–1.75	$0.04522X^{0.9759}$
Molybdenum	0.11–1.74	$0.08204X^{0.9591}$
Nickel	0.10–1.72	$0.05274(X+0.3444)$
Potassium	0.19–1.80	$0.1191X^{0.3661}$
Sodium	0.21–2.03	$0.02916(X+1.8414)$
Silicon	0.17–1.95	$0.07106X^{0.3976}$
Silver	0.08–2.02	$0.07236X^{0.5626}$
Titanium	0.11–1.73	$0.04993X^{0.6002}$
Vanadium	0.10–1.72	$0.06637X^{0.5841}$
Zinc	0.09–1.65	$0.05507X^{0.7402}$

[a] Range of sample means in interlaboratory study.

[b] Where X is the mean concentration, mg/kg.

Reproducibility

Element	Range, mg/kg	Reproducibility, mg/kg
Aluminum	0.25–100	$1.457(X+0.57)^{0.45}$
Barium	28–115	$0.1317X^{.18}$
Boron	0.14–120	$0.9726(X+0.1028)^{0.56}$
Calcium	3.7–11460	$0.2951(X+2.184)$
Chromium	0.18–152	$1.232(X+0.0557)^{0.41}$
Copper	0.47–100	$0.4386(X+0.3459)^{0.85}$
Iron	4.8–210	$0.8323(X+0.0141)^{0.73}$
Lead	0.43–101	$1.814(X+0.6015)^{0.34}$
Magnesium	4.9–1360	$0.3535X$
Manganese	0.3–117	$2.272(X+0.3534)^{0.34}$
Molybdenum	0.21–100	$2.089(X+0.4795)^{0.34}$
Nickel	0.35–100	$1.261(X+0.3238)^{0.40}$
Phosphorus	52–2572	$0.3016(X+14.76)$
Potassium	0.35–247	$1.023(X+0.1154)^{0.63}$
Silicon	3.2–142	$0.8796(X+0.1692)^{0.63}$
Silver	31–102	$0.4439(X+1.2)^{0.88}$
Sodium	3.6–99.6	$0.1075(X+26.36)$
Tin	30–139	$0.7967(X+0.6578)^{0.45}$
Titanium	6.8–103	$0.9682(X+0.9304)^{0.5}$
Vanadium	2.1–101	$1.983(X+0.8418)^{0.41}$
Zinc	5.3–1345	$0.5881(X+1.553)^{0\ 87}$

X = mean concentration in mg/kg.

METAL ANALYSIS IN OILS AND FUELS BY FLAME AAS: D 5863

EXPLANATION

When fuels are combusted, metals present in the fuels can form low forming compounds corrosive to metal parts. Trace metals present in petroleum can deactivate catalysts during processing. These test methods cover the determination of nickel, vanadium, iron, and sodium in crude oils and residual fuels by flame atomic absorption spectrometry.

TEST SUMMARY

Two test methods are available. In Test Method A, the sample is decomposed with sulfuric acid. The residue is digested further with nitric acid, and diluted to volume with dilute nitric acid containing interference suppressant. The metals in the solution are determined by AAS using aqueous matrix matched calibration standards. A nitrous oxide-acetylene flame is used for vanadium, and an air-acetylene flame is used for nickel and iron.

In Test Method B, the sample is diluted with an organic solvent. Interference suppressant is added to the solution if vanadium is to be determined. A nitrous oxide-acetylene flame is used for vanadium, and an air-acetylene flame for nickel and sodium. Oil-soluble metal calibration standards are used. This test method does not purport to quantitatively determine nor detect insoluble particulates.

TEST PRECISION

Element	Range, mg/kg	Test Method	Repeatability, mg/kg	Reproducibility, mg/kg
Vanadium	50–500	A	$1.1X^{0.50}$	$0.33X^{0.90}$
		B	$0.13X^{0.92}$	$1.2X^{0.90}$
Nickel	10–100	A	$0.20X^{0.65}$	$1.3X^{0.53}$
		B	$0.005X^{1.4}$	$0.06X^{1.2}$
Iron	3–10	A	0.98	$1.45X^{1.2}$
Sodium	1–20	B	$0.67X^{0.53}$	$0.67X^{1.0}$

Where X is the mean concentration in mg/kg.

Based on the NIST sample analysis, this test method has no bias for vanadium and nickel in Test Method A and for vanadium in Test Method B. However, nickel, in Test Method B shows a high bias. Bias for iron and sodium is not known.

OPTIMIZATION, CALIBRATION AND VALIDATION OF ICP-AES FOR METAL ANALYSIS: D 7260

This standard practice covers information on the calibration and operational guidance for the multi-element measurements using inductively coupled plasma-atomicemission spectrometry, one of the most widely used technique for such analysis in the oil industry.

CONTAMINANTS IN GAS TURBINE AND DIESEL ENGINE FUELS BY ROTRODE: D 6728

EXPLANATION

During operation of gas turbines and diesel engines, some of the ash-forming substances present in a fuel can lead to high temperature corrosion, as deposition, and fuel system fouling. Fuel contamination specifications vary among the different gas turbine manufacturers. However, without exception, each requires that contaminants must be as low as possible. In most power generation installations, it is the owner's responsibility to verify fuel cleanliness in compliance with turbine manufacturer's warranty specifications. This leads to an on-site analytical instrument performance requirement of below 1.0 mg/kg for several elements. This test method covers such determinations using rotating disk electrode atomic emission spectroscopy.

The test method is applicable to ASTM Grades 0 through 4-GT gas turbine fuels, and Grades Low Sulfur No. 1 and 2-D, and No. 1-D, 2-D, and No. 4-D diesel fuel oils. This test method provides a rapid at-site determination of contamination and corrosive elements ranging from fractions of mg/kg to hundreds of mg/kg in gas turbine and diesel engine fuels so the fuel quality and level of required treatment can be determined. This test method uses oil-soluble metals for calibration and does not purport to quantitatively determine or detect insoluble particles.

TEST METHOD SUMMARY

A fuel test specimen is excited by a controlled arc discharge using the rotating disk technique. The radiant energies of selected analytical lines and a reference are collected and stored by way of photomultiplier tubes, charge coupled devices, or other suitable detectors. A comparison is made of the emitted intensities of the elements in the fuel test specimen against those measured with calibration standards. The concentration of the elements present in the fuel test specimen are calculated and displayed.

INTERFERENCES—There are three kinds of interferences possible in this test method.

(1) Most spectral interferences can be avoided by judicious choice of spectral lines. High concentrations of certain elements can have an interfering influence on the spectral lines used for determining trace levels of contaminants. Instrument manufacturers usually compensate for spectral interferences during factory calibration. A background correction system can also be used for this purpose. If neither of these is effective, the necessary corrections shall be made using the computer software supplied by the instrument manufacturer.

(2) Differences in viscosity of fuel samples will cause differences in uptake rate. Internal references of an instrument will compensate for a portion of the differences. The hydrogen 486.10 nm spectral line shall be used for light fuels, and the carbon 387.10 nm spectral line shall be used as an internal reference to compensate for viscosity effects.

(3) When large particles over 10 μm in size are present in the sample, the analytical results will be lower than the actual concentrations they represent. Large particles may not effectively be transported by the rotating disk electrode sample introduction system into the arc, nor will they be fully vaporized.

TEST PRECISION

The bias was insignificant for light fuels with the exception of calcium, copper, sodium, and zinc at 5 mg/kg concentration, and for heavy fuels, aluminum and nickel at 30 mg/kg.

Repeatability for Light Fuels (ASTM No. 0-GT, No. 1-GT, No. 2-GT, Grade 1-D, Grade 2-D, and Grade 4-D).

Note—X= mean concentration in mg/kg.

Element	ILS Range mg/kg	Repeatability, mg/kg
Aluminum	0.44–10.18	$0.43(X)^{0.33}$
Calcium	0.002–10.2	$0.33(X)^{0.43}$
Chromium	0.34–5.0	$0.31(X)^{0.30}$
Copper	0.05–9.5	$0.36(X)^{0.40}$
Iron	0.14–9.7	$0.53(X)^{0.27}$
Lead	0.18–10.7	$0.74(X+0.02)^{0.33}$
Magnesium	0.021–31.4	$0.29(X+0.01)^{0.61}$
Nickel	0.01–10.0	$0.35(X)^{0.44}$
Potassium	0.02–9.9	$0.38(X)^{0.35}$
Silicon	0.002–9.7	$0.52(X)^{0.48}$
Sodium	0.39–10.4	$0.24(X)^{0.43}$
Vanadium	0.01–16	$0.44(X)^{0.53}$
Zinc	0.07–9.6	$0.34(X)^{0.41}$

Repeatability for Heavy Fuels (ASTM No. 3-GT and No. 4-GT).

Note—X= mean concentration in mg/kg.

Element	ILS Range, mg/kg	Repeatability, mg/kg
Aluminum	0.95–241.0	$0.24(X+0.1)^{0.76}$
Calcium	108–241.8	$0.16(X+0.18)^{0.88}$
Chromium	0.34–226.6	$0.17(X+0.58)^{0.92}$
Copper	0.02–253.0	$0.19(X+0.32)^{0.93}$
Iron	1.04–227.8	$0.34(X)^{0.72}$
Lead	0.30–232.2	$0.72(X+0.32)^{0.54}$
Magnesium	0.86–674.4	$0.16(X)^{0.88}$
Nickel	0.99–235.4	$0.19(X+0.13)^{0.82}$
Potassium	0.37–96.1	$0.33(X)^{0.45}$
Silicon	0.16–228.9	$0.53(X+0.05)^{0.54}$
Sodium	1.14–238.6	$0.14(X)^{0.94}$
Vanadium	1.09–234.4	$0.19(X+0.05)^{0.85}$
Zinc	0.17–166.1	$0.23(X)^{0.87}$

Reproducibility for Light Fuels (ASTM No. 0-GT, No. 1-GT, No. 2-GT, Grade 1-D, Grade 2-D and Grade 4-D).

Note—X= mean concentration in mg/kg.

Element	ILS Range, mg/kg	Repeatability, mg/kg
Aluminum	0.44–10.18	$0.75(X)^{0.33}$
Calcium	0.002–10.2	$0.49(X)^{0.43}$
Chromium	0.34–5.0	$0.48(X)^{0.30}$
Copper	0.05–9.5	$0.66(X)^{0.40}$
Iron	0.14–9.7	$1.16(X)^{0.27}$
Lead	0.18–10.7	$1.50(X+0.02)^{0.33}$
Magnesium	0.021–31.4	$0.77(X+0.01)^{0.61}$
Nickel	0.01–10.0	$0.92(X)^{0.44}$
Potassium	0.02–9.9	$0.61(X)^{0.35}$
Silicon	0.002–9.7	$0.6754(X)^{0.48}$
Sodium	0.39–10.4	$0.48(X)^{0.43}$
Vanadium	0.01–16	$0.70(X+0.01)^{0.53}$
Zinc	0.07–9.6	$0.51(X)^{0.41}$

Reproducibility for Heavy Fuels (ASTM No. 3-GT and No. 4-GT).
NOTE—X= mean concentration in mg/kg.

Element	ILS Range, mg/kg	Repeatability, mg/kg
Aluminum	0.95–24	$0.40(X+0.07)^{0.767}$
Calcium	1.08–241.8	$0.2958(X+0.18)^{0.88}$
Chromium	0.34–226.6	$0.31(X+0.58)^{0.92}$
Copper	0.02–253.0	$0.285(X+0.32)^{0.93}$
Iron	1.04–227.8	$0.67(X)^{0.723}$
Lead	0.30–232.2	$1.33(X+0.32)^{0.54}$
Magnesium	0.86–674.4	$0.31(X)^{0\ 88}$
Nickel	0.99–235.4	$0.52(X+0.13)^{0\ 825}$
Potassium	0.37–96.1	$0.65(X)^{0.45}$
Silicon	0.16–228.9	$0.88(X+0.1)^{0.54}$
Sodium	1.14–238.6	$0.32(X)^{0.94}$
Vanadium	1.09–234.4	$0.55(X+0.05)^{0\ 85}$
Zinc	0.17–166.1	$0.41(X+0)^{0\ 87}$

METAL ANALYSIS INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY: D 4951

TEST SUMMARY

This test method is applicable for the determination of boron, copper, sulfur, phosphorus, calcium, magnesium, and zinc in lubricating oils and additives. Higher or lower concentrations can be determined by using additional and appropriate dilution.

A sample is weighed and then diluted with mixed xylenes or other suitable solvent on a weight-by-weight basis. Standards are prepared in the same manner. An internal standard must be added to the solutions to test for variations in sample-solution introduction. The solutions are introduced by using a peristaltic pump or free aspiration to an inductively coupled plasma (ICP) and elemental determinations are made by comparing standard and sample atomic emission intensities at wavelengths associated with the desired elements.

Significant under-recoveries are obtained when VI improvers are present in the oil sample. This has been corroborated by several oil company laboratories. To overcome this interference, dilute the sample >40 fold and use cobalt or other internal standard.

TEST PRECISION[1]

Repeatability.

Element	Range, mass%	Sample	Repeatability, mass %
Ba	0.13	Oil	0.011
Ba	3.4	Additive	0.20
B	0.01–0.02	Oil	0.0017
B	0.11–0.13	Additive	0.0093
Ca	0.012–0.18	Oil	$0.0145(X+0.152)^{0.67}$
Ca	0.8–4.1	Additive	$0.0363X$
Cu	0.01–0.02	Oil	0.0008
Cu	0.11	Additive	0.0054
Mg	0.05–0.14	Oil	$0.0159X^{0.7}$
Mg	0.35–0.82	Additive	$0.0473X$
Mo	0.005–0.05	Oil	$0.002175(X0.4863)$
Mo	0.043–0.50	Additive	$0.01114(X0.8645)$
P	0.05–0.12	Oil	$0.0264X$
P	0.7–1.3	Additive	$0.0313(X+0.294)$
S	0.3–0.8	Oil	0.016

S	3.0–3.2	Additive	0.14
Zn	0.05–0.13	Oil	$0.0212(X+0.0041)$
Zn	0.7–1.4	Additive	0.035

NOTE—X = mean concentration, mass %.

Reproducibility.

Element	Range, mass %	Sample	Reproducibility, mass %
Ba	0.13	Oil	0.019
Ba	3.4	Additive	0.66
B	0.01–0.02	Oil	0.0035
B	0.11–0.13	Additive	0.016
Ca	0.012–0.18	Oil	$0.0280(X+0.152)^{0.67}$
Ca	0.8–4.1	Additive	$0.114X$
Cu	0.01–0.02	Oil	0.0017
Cu	0.11	Additive	0.016
Mg	0.05–0.14	Oil	$0.0624X^{0.7}$
Mg	0.35–0.82	Additive	$0.198X$
Mo	0.005–0.05	Oil	$0.005704(X0.4863)$
Mo	0.043–0.50	Additive	$0.05006(X0.8645)$
P	0.05–0.12	Oil	$0.101X$
P	0.7–1.3	Additive	$0.115(X+0.294)$
S	0.3–0.8	Oil	0.061
S	3.0–3.2	Additive	0.372
Zn	0.05–0.13	Oil	$0.0694(X+0.0041)$
Zn	0.7–1.4	Additive	0.115

NOTE—X = mean concentration, mass %.

[1] These tables are Tables 2 and 3 from Test Method D 4951.

METAL ANALYSIS INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY:

D 5185

EXPLANATION

Costs associated with damages due to engine and machine wear can be very significant. Thus, diagnostic methods for determining the condition of an engine or other equipment in service are important. This test method, which is a diagnostic method that indicates the condition of oil-wetted components in engines, represents the only test method for the quantitative determination of metals in used oils. This test method covers the determination of additive elements, wear metals, and contaminants in used lubricating oils by inductively coupled plasma atomic emission spectrometry (ICPAES). It can also be used for unused oils to provide more complete elemental composition data than Test Methods D 4628, D 4927, or D 4951.

TEST SUMMARY

A weighed portion of a thoroughly homogenized used oil is diluted 10-fold by weight with mixed xylenes or other suitable solvent. Standards are prepared in the same manner. An optional internal standard can be added to the solutions to compensate for variations in sample introduction efficiency. The solutions are introduced to the ICP instrument by free aspiration or an optional peristaltic pump. By comparing emission intensities of elements in the specimen with emission intensities measured with the standards, the concentrations of elements in the specimen are calculable.

TEST PRECISION

Element	Range, μg	Repeatability, μg/g	Reproducibility, μg/g
Aluminum	1–40	$0.71X^{0.4}$	$3.8X^{0.3}$
Barium	0.2–4	$0.24X^{0.7}$	$0.59X^{0.9}$
Boron	0.7–30	$0.26X$	$13X^{0.01}$
Calcium	40–9000	$0.0020X^{1.4}$	$0.015X^{1.3}$
Chromium	0.7–40	$0.17X^{0.8}$	$0.81X^{0.6}$
Copper	2–160	$0.12X^{0.9}$	$0.24X$
Iron	2–140	$0.13X^{0.8}$	$0.52X^{0.8}$
Lead	0.5–160	$1.6X^{0.3}$	$3.0X^{0.4}$
Magnesium	5–1700	$0.16X^{0.9}$	$0.72X^{0.8}$
Manganese	5–700	$0.010X^{1.3}$	$0.13X^{1.2}$
Molybdenum	1–200	$0.29X^{0.7}$	$0.64X^{0.7}$
Nickel	0.4–40	$0.52X^{0.5}$	$1.5X^{0.5}$
Phosphorous	2–1000	$1.3X^{0.6}$	$4.3X^{0.5}$
Potassium	0.3–1200	$3.8X^{0.3}$	$6.6X^{0.3}$
Silicon	2–50	$1.3X^{0.3}$	$2.9X^{0.4}$
Silver	0.3–50	$0.15X^{0.8}$	$0.35X$
Sodium	2–70	$0.49X^{0.7}$	$1.1X^{0.7}$
Sulfur	900–6000	$0.49X^{0.8}$	$1.2X^{0.7}$
Tin	3–40	$2.4X^{0.2}$	$2.1X^{0.6}$
Titanium	0.1–40	$0.54X^{0.4}$	$2.5X^{0.5}$
Vanadium	1–50	$0.061X$	$0.28X^{1.1}$
Zinc	60–1600	$0.15X^{0.9}$	$0.083X^{1.1}$

Where X = mean concentration, μg/g.

METALS IN LUBRICATING GREASES BY ICP-AES: D 7303

EXPLANATION

Lubricating greases are used in almost all bearings used in any machinery. Lubricating greases are composed of about 90 % additized oil and soap or other thickening agent. There are over a dozen metallic elements present in grease, either blended as additives for performance enhancements or as thickeners, or in used grease present as contaminants and wear metals. Determination of their concentration can be an important aspect of grease manufacture. The metal content can also indicate the amount of thickeners in the grease.

This is the first industry standard available for simultaneous multi-element analysis of lubricating greases. The metals determined by this method include Al, Sb, Ba, Ca, Fe, Li, Mg, Mo, P, Si, Na, S, and Zn. It may also be possible to determine other elements such as Bi, B, Cd, Cr, Cu, Pb, Mn, K, Ti, etc., by this technique.

TEST SUMMARY

A weighed portion of the grease sample subjected to alternate means of sample dissolution which may include sulfated ashing in a muffle furnace or by closed vessel microwave digestion in acid. Ultimately, these diluted acid solutions are analyzed using ICP-AES. Aqueous calibration standards are used. The solutions are introduced to the ICP instrument by free aspiration or an optional peristaltic pump.

Interferences—Spectral interferences can usually avoided by judicious choice of analytical wavelengths. If the grease sample contains refractory additives such as Si or Mo, it is possible that some of these elements may remain undissolved in the residue, and may result in lower recoveries. If HF is used for dissolution of grease residues, elements such as silicon may be lost as SiF_6. Residual HF can also attack the ICP sample introduction system. HF can be passivated by adding dilute boric acid to the acid solution. If the dry ashing in sample preparation step is used, elements such as sulfur will be volatilized during combustion.

TEST PRECISION[1]

[1] This table is Table 1 in Test Method 07303-6.

Element	Range, mg/kg	Repeatability	Reproducibility
Aluminum	10–600	$0.2163\ X^{0.9}$	$6.8156\ X^{0.9}$
Antimony	10–2300	$0.3051\ X^{0\ 8191}$	$4.6809\ X^{0\ 8191}$
Barium	50–800	$0.3165\ X^{0.7528}$	$2.9503\ X^{0.7528}$
Calcium	20–50 000	$2.2853\ X^{0.7067}$	$3.0571\ X^{0.7067}$
Iron	10–360	$0.8808\ X^{0.7475}$	$2.5737\ X^{0.7475}$
Lithium	300–3200	$0.0720\ X^{1.0352}$	$1.1476\ X^{1.0352}$
Magnesium	30–10 000	$0.6620\ X^{0.6813}$	$2.6155\ X^{0.6813}$
Molybdenum	50–22 000	$0.1731\ X^{0.9474}$	$0.4717\ X^{0.9474}$
Phosphorus	50–2000	$1.2465\ X^{0.6740}$	$4.0758\ X^{0.6740}$
Silicon	10–15 000	$1.3859\ X^{0.9935}$	$4.8099\ X^{0.9935}$
Sodium	30–1500	$6.5760\ X^{0.5}$	$11.571\ X^{0.5}$
Sulfur	1600–28 000	$1.0507\ X^{0\ 8588}$	$1.5743\ X^{0\ 8588}$
Zinc	300–2200	$0.1904\ X^{0\ 8607}$	$0.5912\ X^{0\ 8607}$

NOTE—X= mean concentration in mg/kg.

METAL ANALYSIS IN CRUDE OILS AND FUELS BY ICP-AES:

D 5708

EXPLANATION

When fuels are combusted, vanadium present in the fuel can form corrosive compounds. The value of crude oils can be determined, in part, by the concentrations of nickel, vanadium, and iron. Nickel and vanadium present at trace levels in petroleum fractions can deactivate catalysts during processing. These tests provide a means of determining vanadium, nickel, and iron, in crude oils and residual fuels by inductively coupled plasma atomic emission spectrometry. These test methods complement Test Method D 1548, which determines vanadium by AAS and Test Method D 5863, which determines the same elements by AAS. Typically lower limits of quantitation by these test methods are a few tenths of mg/kg.

TEST SUMMARY

Test Method A—The sample is dissolved in an organic solvent to make a 10 % m/m sample solution. This is analyzed by ICPAES using organometallic calibration standards.

Test Method B—The sample is decomposed with sulfuric acid. The residual carbon is burned off in a muffle furnace, and the inorganic residue is dissolved in dilute nitric acid. This solution is analyzed by ICPAES using aqueous calibration standards.

TEST PRECISION

Element	Range, mg/kg	Test Method	Repeatability, mg/kg	Reproducibility, mg/kg
Vanadium	50–500	A	$0.070X^{0.88}$	$0.12X^{1.1}$
		B	$0.02X^{1.1}$	$0.10X^{1.1}$
Nickel	10–100	A	$0.01X^{1.3}$	$0.41X^{0.78}$
		B	$0.02X^{1.2}$	$0.05X^{1.3}$
Iron	1–10	A	$0.22X^{0.30}$	$0.68X^{0.36}$
		B	$0.23X^{0.67}$	$0.91X^{0.51}$

Based on the analysis of NIST samples, there was no bias for both of these test methods for vanadium and nickel. The test for iron could not be determined.

PHOSPHORUS IN ILSAC GF4 ENGINE OILS BY ICP-AES: D 7040

EXPLANATION

GF 4 grade engine oils marketed in the year 2004–2005 and beyond will have a maximum phosphorus content of 500 to 800 mg/kg. These limits are required to minimize poisoning of automotive emission control catalysts by volatile phosphorus species. It is anticipated that the later grades of oils may have even lower levels of phosphorus. This test method provides more precise results than other similar elemental analysis methods D 4927 WDXRF, and D 4951 or D 5185 ICP-AES methods. The precision statements given in this method are valid for dilutions in which the mass % of sample in solvent is held constant in the range of 1 to 5 mass % oil. Although the precision tables in this test method define the concentration range of 500 to 800 mg/kg phosphorus, both higher and lower concentrations of phosphorus can be determined by this method.

TEST SUMMARY

A sample portion is weighed and diluted by mass with mixed xylenes or other solvent. An internal standard, which is required, is either weighed separately into the test solution or is previously combined with the dilution solvent. Calibration standards are similarly prepared. The solutions are introduced to the ICP instrument by a peristaltic pump (required). By comparing emission intensity of phosphorus in the test specimen with emission intensities measured with the calibration standards and by applying the appropriate internal standard and background corrections, the concentrations of phosphorus in the sample is calculated.

INTERFERENCES—There are no known spectral interferences between phosphorus and other elements covered by this method when using the spectral lines 177.51, 178.29, 185.94, 213.62, 214.91, or 253.40 nm for phosphorus. However, if spectral interferences exist because of other interfering elements or selection of other spectral lines, correct for the interference using the technique described in the Test Method D 5185. Viscosity index improvers, which may be present in the multi-grade lubricating oils, can bias the measurements. However, the biases can be reduced to negligible proportions by using the specified solvent-to-sample dilution and an internal standard.

TEST PRECISION

Repeatability:	$0.00317X^{1.32}$
Reproducibility:	$0.01115X^{1.32}$

Where X is the mean concentration of phosphorus in mg/kg.

The bias of this method cannot be determined since there are no accepted standard reference materials with certified values for phosphorus in these engine oils.

METAL ANALYSIS IN PETROLEUM COKE BY AAS: D 5056

EXPLANATION

The presence and concentration of various metallic elements in a petroleum coke are major factors in the suitability of the coke for various uses. This test method can be used by the buyer and seller in the commercial transfer of petroleum coke to determine whether that lot of coke meets the specifications of the purchaser. This method can analyze raw and calcined coke for trace elements aluminum, calcium, iron, nickel, silicon, sodium, and vanadium. See complementary ICPAES method for the same analysis for Test Method D 5600 that follows this test method analysis.

TEST SUMMARY

A sample of petroleum coke is ashed at 525°C. The ash is fused with lithium tetra- or meta-borate. The melt is dissolved in dilute hydrochloric acid and the resultant solution is analyzed by AAS. Spectral interferences may occur when using wavelengths other than those recommended for analysis, or when using multi-element hollow cathode lamps.

TEST PRECISION[1]

Repeatability and Reproducibility Ranges.

Metal, Range mg/kg	Repeatability	Reproducibility
Aluminum, 15 to 105	15	40
Calcium, 20 to 225	21	36
Iron, 150 to 500	$0.39X^{3/4}$	$1.18X^{3/4}$
Nickel, 5 to 200	$1.27X^{1/2}$	$1.69X^{1/2}$
Silicon, 90 to 420	$1.94X^{1/2}$	$9.41X^{1/2}$
Sodium, 15 to 115	$0.19X$	$0.61X$
Vanadium, 5 to 500	$1.19X^{1/2}$	$2.93X^{1/2}$

NOTE—X = The average value of two results in parts per million (milligrams per kilogram).

[1] This table is Table 3 from Test Method D 5056.

The bias of this test method has not been determined.

METAL ANALYSIS IN PETROLEUM COKE BY ICPAES: **D 5600**

EXPLANATION

See Test Method D 5056 just previous to this analysis.

TEST SUMMARY

A sample of petroleum coke is ashed at 700°C. The ash is fused with lithium borate. The melt is dissolved in dilute hydrochloric acid, and the resultant solution is analyzed by ICPAES using aqueous calibration standards.

TEST PRECISION[1]

Repeatability and Reproducibility.

Metal, mg/kg	Repeatability	Reproducibility
Aluminum	$0.32\ X^{0.66}$	$0.92\ X^{0.66}$
Barium	$0.19\ X^{0.66}$	$0.71\ X^{0.66}$
Calcium	7.2	20.8
Iron	$1.66\ X^{0.5}$	$3.77\ X^{0.5}$
Magnesium	$0.21\ X^{0.66}$	$0.61\ X^{0.66}$
Manganese	$0.042\ (X+2.55)$	$0.34\ (X+2.55)$
Nickel	$0.52\ X^{0.66}$	$0.96\ X^{0.66}$
Silicon	$0.71\ (X+4.80)$	$0.20\ (X+4.80)$
Sodium	$1.04\ X^{0.5}$	$3.52\ X^{0.5}$
Titanium	0.75	1.16
Vanadium	$0.20\ X^{0.75}$	$0.35\ X^{0.75}$
Zinc	$1.07\ X^{0.33}$	$2.20\ X^{0.33}$

NOTE—X = the average value of two results in mg/kg,

The bias for this test method has not been determined.

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[1] This table is Table 2 from Test Method D 5600-04e1.

METAL ANALYSIS IN PETROLEUM COKE BY WAVELENGTH DISPERSIVE X-RAY SPECTROSCOPY: **D 6376**

EXPLANATION

The presence and concentration of sulfur and various metallic elements in a petroleum coke are major factors determining the suitability of a coke for various purposes. This test method provides a rapid means of measuring these elements in coke samples, and provides a guide for determining conformance to material specifications used by buyers and sellers in commercial transfer of petroleum coke. Sulfur content can also be used to evaluate potential formation of sulfur oxides, a source of atmospheric pollution. This test method using X-ray fluorescence spectrometry specifically determines sodium, aluminum, silicon, sulfur, calcium, titanium, vanadium, manganese, iron, and nickel. This test method may be applicable to additional elements if appropriate standards are available.

Appropriate matrix and interelement spectral interferences need to be corrected to obtain accurate results.

TEST SUMMARY

A weighed portion of a sample of petroleum coke dried at 110°C and crushed to pass a 200-mesh sieve is mixed with stearic acid and then milled and compressed into a smooth pellet. The pellet is then irradiated with an X-ray beam. The characteristic X-rays of the elements analyzed are excited, separated and detected by the wavelength dispersive X-ray spectrometer. The measured X-ray intensities are converted to elemental concentration by using a calibration equation derived from the analysis of the standard materials. The *K*-alpha spectral lines are used for all of the elements determined by this test method.

TEST PRECISION

The precision and bias of this test method are under development at present.

WEAR METALS AND CONTAMINANTS IN USED OILS/ HYDRAULIC FLUIDS USING ROTRODE EMISSION SPECTROMETRY:

D 6595

EXPLANATION

The determination of debris in used lubricating oils is a key diagnostic method practiced in machine condition monitoring programs. The presence or increase in concentration of specific wear metals can be indicative of the early stages of wear if there are baseline concentration data for comparison. A marked increase in contaminant element can be indicative of foreign materials in the lubricants, such as antifreeze or sand, which may lead to wear or lubricant degradation. The test method identifies the metal and their concentration so that trends relative to time or distance can be established and corrective action can be taken prior to more serious or catastrophic failure.

This test method uses oil-soluble metals for calibration and does not purport to relate quantitatively the values determined as insoluble particles to the dissolved metals. Analytical results are particle size dependent and low results may be obtained for those elements present in used oil samples as large particles.

TEST SUMMARY

Wear metals and contaminants in a used oil test specimen are evaporated and excited by a controlled arc discharge using the rotating disk technique. The radiant energies of selected analytical lines and one or more references are collected and stored by way of photomultiplier tubes, charge coupled devices, or other suitable detectors. A comparison is made of the emitted intensities of the elements in the used oil test specimen against those measured with calibration standards. The concentrations of the elements present in the oil test specimen are calculated, displayed, or entered into a database for processing.

INTERFERENCES—There could be three types of interferences in this method. Refer to Test Method D 6728 for discussion on interferences.

TEST PRECISION

Bias was insignificant for all elements with the exception of barium at 30 mg/kg, nickel at 50 mg/kg, and titanium at 100 mg/kg concentrations. The calculated bias values are of lesser significance in used oil trending applications.

Test Precision			
Element	Range, mg/kg	Repeatability, mg/kg	Reproducibility, mg/kg
Aluminum	0.23–101	$0.5419(X+0.57)^{0.45}$	$1.457(X+0.57)^{0.45}$
Barium	28–115	$0.0694X^{1.18}$	$0.1317X^{.18}$
Boron	0.14–120	$0.4280(X+1028)^{0.56}$	$0.9726(X+0.1028)^{0.56}$
Calcium	3.7–11460	$0.1106(X+2.184)$	$0.2951(X+2.184)$
Chromium	0.18–152	$0.7285(X+0.0557)^{0.41}$	$1.232(X+0.0557)^{0.41}$
Copper	0.47–100	$0.1631(X+0.3459)^{0.85}$	$0.4386(X+0.3459)^{0.85}$
Iron	4.8–210	$0.3159(X+0.014)^{0.73}$	$0.8323(X+0.0141)^{0.73}$
Lead	0.43–101	$1.062(X+0.6015)^{0.34}$	$1.814(X+0.6015)^{0.34}$
Magnesium	4.9–1360	$0.1049X$	$0.3535X$
Manganese	0.3–117	$0.7017(X+0.3534)^{0.34}$	$2.272(X+0.3534)^{0.34}$
Molybdenum	0.21–100	$0.9978(X+0.4795)^{0.34}$	$2.089(X+0.4795)^{0.34}$
Nickel	0.35–100	$0.7142(X+0.3238)^{0.40}$	$1.261(X+0.3238)^{0.40}$
Phosphorus	52–2572	$0.0761(X+14.76)$	$0.3016(X+14.76)$
Potassium	0.35–247	$0.4075(X+0.1154)^{0.63}$	$1.023(X+0.1154)^{0.63}$
Silicon	3.2–142	$0.4015(X+0.1692)^{0.63}$	$0.8796(X+0.1692)^{0.63}$
Silver	31–102	$0.1523(X+1.2)^{0.88}$	$0.4439(X+1.2)^{0.88}$
Sodium	3.6–99.6	$0.1231(X-2.674)$	$0.1075(X+26.36)$
Tin	30–139	$0.6777(X+0.9304)^{0.5}$	$0.7967(X+0.6578)^{0.45}$
Titanium	6.8–103	$0.5831(X+0.9304)^{0.5}$	$0.9682(X+0.9304)^{0.5}$
Vanadium	2.1–101	$0.6389(X+0.8418)^{0.41}$	$1.983(X+0.8418)^{0.41}$
Zinc	5.3–1345	$0.2031(X+1.553)^{0.87}$	$0.5881(X+1.553)^{0\ 87}$

X = mean concentration in mg/kg.

METAL ANALYSIS X-RAY FLUORESCENCE SPECTROMETRY:

D 4927

(Equivalent Test Methods: IP 407 and DIN 51391 T2)

TEST SUMMARY

This test method covers the determination of barium, calcium, phosphorus, sulfur, and zinc in unused lubricating oils at element concentration ranges of 0.03 to 1.0 % mass (0.01 to 2.0 % for sulfur). The range may be extended to higher concentrations by dilution of sample specimens. Additives may also be determined after dilution with base oil. Two different procedures are presented in this test method:

Procedure A: Internal Standard Procedure—Internalstandards are used to compensate for inter-element effects of X-ray excitation and fluorescence.

Procedure B: Mathematical Correction Procedure—The measured X-ray fluorescence intensity for a given element is mathematically corrected for potential interference from other elements present in the sample.

A sample specimen is placed in the X-ray beam and the intensity of the appropriate fluorescence lines of barium, calcium, phosphorus, sulfur, and zinc are measured. Instrument response factors related to the concentration of standards enable the determination of the concentration of elements in the tested sample specimens. Enhancement or depression of the X-ray fluorescence of a given element by an interfering element in the sample may occur. This is corrected by internal standards in Procedure A, and by mathematical correction in Procedure B.

TEST PRECISION

Repeatability and Reproducibility.

Element	Repeatability	Reproducibility
	Internal Standard Procedure	
Barium	$0.03214\ X^{1.059}$	$0.07105\ X^{1.059}$
Calcium	$0.0285(X+0.003)^{0.77}$	$0.078(X+0.003)^{0.77}$
Phosphorus	$0.0411\ X^{0.756}$	$0.078\ X^{0.756}$
Sulfur	$0.03966\ X$	$0.2098\ X$
Zinc	$0.0157\ X^{0.83}$	$0.0373\ X^{0.83}$
	Mathematical Correction Procedure	
Barium	$0.02028\ X$	$0.1593\ X$
Calcium	$0.018\ X^{0.71}$	$0.07841\ X^{0.71}$
Phosphorus	$0.033\ X^{0.812}$	$0.1138\ X^{0.812}$
Sulfur	$0.05335(X+0.001)$	$0.1669(X+0.001)$
Zinc	$0.0197(X+0.001)^{1.114}$	$0.1206(X+0.001)^{1.114}$

METAL ANALYSIS IN LUBRICATING OILS AND ADDITIVES BY WAVELENGTH DISPERSIVE X-RAY FLUORESCENCE (WD-XRF):

D 6443

TEST SUMMARY

This test method covers the determination of additive elements calcium, chlorine, copper, magnesium, phosphorus, silicon, and zinc in unused lubricating oils, additives, and additive packages. Matrix effects are handled with mathematical corrections. This test method uses regression software to determine calibration parameters, which can include interelement effect coefficients, usually referred to as alphas. Alphas can also be determined from theory using relevant software.

The X-ray spectrometer is initially calibrated for each element by determining the slope and intercept of the calibration curve by regressing concentration data and intensities measured on a set of physical standards. Theoretical alphas, or a combination of theoretical and empirical alphas can also be used. A sample is placed in the X-ray beam and the intensities of the appropriate fluorescence lines are measured. A similar measurement is made at an offset wavelength to obtain a background correction. Enhancement or absorption of the X-rays can occur, and these effects can be handled in the data reduction by implementation of alphas. Concentrations of the analytes are determined by comparison of net signals against calibration curves.

TEST PRECISION[1]

See the following tables for precision estimates for lubricating oils and additives.

Repeatability and Reproducibility for Oils (Units are Mass Percent).

Analyte	Concns	Repeatability	Reproducibility
Ca	0.001–0.200	$0.006914\ (X+0.0007)^{0.5}$	$0.04762\ (X+0.0007)^{0.5}$
Cl	0.001–0.030	$0.0356\ (X+0.0086)$	$0.05612\ (X+0.0340)$
Cu	0.001–0.030	$0.002267\ (X+0.0013)^{0.4}$	$0.01068\ (X+0.0013)^{0.4}$
Mg	0.003–0.200	$0.01611\ (X+0.0008)^{0.333}$	$0.05208\ (X+0.0008)^{0.333}$
P	0.001–0.200	$0.02114\ X^{0.7}$	$0.09112\ X^{0.7}$
S	0.030–0.800	$0.02371\ X^{0.9}$	$0.1623\ X^{0.9}$
Zn	0.001–0.200	$0.01225\ X^{0.7}$	$0.06736\ X^{0.7}$

NOTE—X= concentration in mass %.

Analyte	Concn(s)	Repeatability	Reproducibility
Ca	1.00–1.50	0.0226	0.1151
Cl	0.070	0.0039	0.0104
Mg	0.30–1.00	0.0721	0.1797
P	0.30–1.50	$0.02448\ X^{0.8}$	$0.1663\ X^{0.8}$
S	1.00–5.00	$0.02783\ X^{0.8}$	$0.1744\ X^{0.8}$
Zn	0.30–1.50	$0.02002\ X^{1.08}$	$0.1183\ X^{1.08}$

NOTE—X = concentration in mass %.

[1] These tables are Tables 4 and 6 from Test Method D 6443.

The bias was found to be not significant based on the analysis of synthetically prepared oils.

METALS IN LUBRICATING OILS BY ENERGY DISPERSIVE X-RAY FLUORESCENCE SPECTROSCOPY: D 6481

EXPLANATION

Some oils are formulated with organo-metallic additives, which act, for example, as detergents, antioxidants, and antiwear agents. Some of these additives contain one or more of these elements: calcium, phosphorus, sulfur, and zinc. This test method provides a means of determining the concentrations of these elements, which in turn provide an indication of the additive content of these oils. This test method uses interelement correction factors calculated from empirical calibration data. This test method is not suitable for the determination of magnesium and copper at the concentrations present in lubricating oils. The method also does not cover lubricating oils that contain chlorine or barium as an additive element. This test method can be used by persons who are not skilled in X-ray spectrometry. It is intended to be used as a routine test method for production control analysis.

TEST SUMMARY

A specimen is placed in the X-ray beam, and the appropriate regions of the spectrum are measured to give the fluorescent intensities of elements of interest. Other regions of the spectrum are measured to compensate for varying background. If the detector does not completely resolve all the elements in a single measurement, then to improve selectivity, there is a combination of sequential and simultaneous measurements employing primary and secondary beam filters. There can be correction of measured intensities for spectral overlap. Concentrations of elements of interest are determined by comparison of these intensities against a calibration curve using empirical interelement correction factors and ratio to backscatter. The EDXRF spectrometer is initially calibrated using a set of prepared standards to collect the necessary intensity data. Each calibration line and any correction coefficient are obtained by a regression of these data, using the program supplied with the spectrometer.

INTERFERENCES—The additive elements found in lubricating oils will affect the measured intensities from the elements of interest to a varying degree. In general, for lubricating oils, the X-radiation emitted by the elements of interest can be absorbed by itself (self-absorption) or by the other elements present in the sample matrix. Also, the X-radiation emitted from one element can further excite (enhance) another element. These interelement effects are significant at concentrations varying from 0.03 mass % 5, due to the heavier elements, to 1 mass %, for the lighter elements. Enhancement effects can be minimized by selective excitation. The measured concentration for a given element can be mathematically corrected for self-absorption and for interelement effects by other elements present in the sample matrix. If an element is present at significant concentrations and an interelement correction for that element is not employed, the results can be low due to absorption or high due to enhancement.

If a sample containing barium as an additive above 0.03 mass % is measured against a calibration derived from standards without barium, then results will be low. If a sample containing chlorine as an impurity above 0.03 mass % is measured against a calibration derived from standards without chlorine, then the results can be affected. There can be spectral overlap of one elements onto another, especially for phosphorus on sulfur, and the instrument must include correction procedures for any such overlaps.

TEST PRECISION

Element	Repeatability, mass %	Reproducibility, mass %
Phosphorus	0.0060	0.0199
Sulfur	$0.01648(X+0.0141)^{0.8}$	$0.1024(X+0.0141)^{0.8}$
Calcium	$0.006795(X+0.0120)^{0.5}$	$0.1492(X+0.0120)^{0.5}$
Zinc	$0.003274X^{0.25}$	$0.02165X^{0.25}$

Where X is the concentration of element in mass %.

No information can be presented on the bias of this procedure since no material having an accepted reference value is available.

METHANOL IN CRUDE OILS BY MULTI-DIMENSIONAL GAS CHROMATOGRAPHY:

D 7059

EXPLANATION

Methanol is used in production of crude oils to prevent formation of gas hydrates. The presence of residual methanol in crude oils can lead to costly problems in refinery operations. This method uses direct injection multidimensional gas chromatography in the concentration range of 15–900 ppm (m/m). The pooled limit of quantitation is 15 ppm (m/m).This test method is applicable only to crude oils containing less than or equal –0.1 % (v/v) water. This test method has not been tested with crude oils samples that are solid or waxy, or both, at ambient temperatures.

TEST SUMMARY

An internal standard, 1-propanol, is added to the sample which is then introduced into a gas chromatograph equipped with two columns and a flow switching system between the two columns. The sample first passes through the polydimethylsiloxane WCOT column that performs a pre-separation of the methanol and 1-propanol and eliminates unwanted hydrocarbons. The methanol and 1-propanol are transferred to the analytical CP-Lowox column. While the methanol and 1-propanol are eluting from the CP-Lowox analytical column, auxiliary carrier gas is used to elute higher boiling crude oil hydrocarbons from the pre-column, either in the forward or backflush mode, to yield a stable baseline for the next analysis.

TEST PRECISION

Repeatability:	$0.063(X+60)$
Reproducibility:	$0.107(X+60)$

Where X is the mean value of replicates. The applicable range for above precision is 15 to 900 ppm (m/m).

The bias of this method cannot be determined because no material having an accepted reference value is currently available.

TRACE METHANOL IN PROPYLENE CONCENTRATES BY GAS CHROMATOGRAPHY:

D 4864

EXPLANATION

Methanol is a common impurity in propylene. It can have a deleterious effect on various processes that use propylene as a feedstock. The range of this method is approximately 4 to 40 mg/kg.

TEST SUMMARY

A known weight of water is pressured into a sample cylinder containing a known amount of liquefied propylene. The contents in the cylinder are shaken and the water/methanol phase is withdrawn. A reproducible volume of the extract is then injected into a gas chromatograph equipped with either a thermal conductivity or a flame ionization detector. The methanol concentration is calculated from the area of the methanol peak using calibration and extraction factors obtained from synthetic blends of known methanol content.

There are no interferences using the GC columns referenced in this test method. However, any water-soluble component that co-elutes with methanol on any other GC column used would interfere.

TEST PRECISION

Repeatability:	2.4
Reproducibility:	8.0

Above precision values are expressed in terms of the maximum allowable ratio of the larger to the smaller result. Since there is no accepted reference material suitable for determining the bias for this procedure, the bias is not available.

MOLECULAR WEIGHT OF LUBRICATING OILS: D 2878

EXPLANATION

This test method provides a calculation routine for converting data obtained by Test Method D 972 to apparent vapor pressure and molecular weight. This test method is applicable to petroleum based and synthetic ester lubricating oils at temperatures of 250 to 500°F. Its applicability to lubricating greases has not been established.

TEST SUMMARY

The test is run at the selected temperature for a sufficient time to give the selected amount of evaporation, which is 5 ± 1 % unless otherwise specified. This evaporation rate is compared with a standard value for pure *m*-terphenyl to yield the apparent vapor pressure and molecular weight of the lubricating oil.

TEST PRECISION

No independent precision or bias statement specific for this test method can be issued at this time. However, based on evaporation loss, Test Methods D 972 and D 2595, the following precision may be expected:

	Repeatability	Reproducibility
D 972	6 %	23 %
D 2595	23 %	35 %

MOLECULAR WEIGHT OF HYDROCARBONS: D 2503

EXPLANATION

Molecular weight is a fundamental physical constant that can be used in conjunction with other physical properties to characterize pure hydrocarbons and their mixtures. A knowledge of molecular weight is necessary for the application of a number of correlative methods that are useful in determining the gross composition of the heavier fractions of petroleum. This test method can be applied to petroleum fractions with molecular weights up to 3000; however, the precision of this test method has not been established beyond 800 molecular weight. This test method should not be applied to oils having initial boiling points lower than 220°C.

TEST SUMMARY

The sample is dissolved in an appropriate solvent. A drop each of this solution and the solvent are suspended, side by side, on separate thermistors in a closed chamber saturated with solvent vapor. The solvent condenses on the sample drop and causes a temperature difference between the two drops. The resultant change in temperature is measured and used to determine the molecular weight of the sample by reference to a previously prepared calibration curve.

TEST PRECISION

Molecular Weight Range	Repeatability, g/mol	Reproducibility, g/mol
245 to 399	5	14
400 to 599	12	32
600 to 800	30	94

Bias for this test method has not been established.

MOLECULAR WEIGHT OF PETROLEUM OILS:
D 2502

EXPLANATION

This test method calculates molecular weight of petroleum oils from kinematic viscosity measurements at 100 and 210°F. It is applicable to samples with molecular weights in the range from 250 to 700 and is intended for use with average petroleum fractions. It should not be applied indiscriminately for oils that represent extremes of composition or possess an exceptionally narrow molecular weight range.

TEST SUMMARY

Kinematic viscosity (Test Method D 445) of the sample is measured at 100 and 210°F, and then using the tables provided, the molecular weight is calculated.

TEST PRECISION

This test method has a repeatability of 3 and a reproducibility of 25 g/mol.

The bias is not known.

MOISTURE CORROSION RESISTANCE OF AUTOMOTIVE GEAR LUBRICANTS:
D 7038

EXPLANATION

This test simulates a type of severe field service in which corrosion-promoting moisture in the form of condensed water vapor accumulates in the axle assembly. This may happen as a result of volume expansion and contraction of the axle lubricant and the accompanied breathing in of moisture-laden air through the axle vent. This test screens the lubricants for their ability to prevent the expected corrosion. ASTM Test Monitoring Center (TMC) provides reference oils and an assessment of the test results obtained on those oils by the laboratory. In addition to helping a lab assess its capability versus that of others in the industry, various agencies require that a laboratory utilize the TMC services in seeking qualification of oils against specifications.

This test method uses a bench-mounted hypoid differential housing assembly. This test method is commonly referred to as L-33-1 test.

TEST SUMMARY

This procedure utilizes a Dana Corporation model 30 hypoid differential assembly, 4:10 ratio, standard differential with uncoated ring and pinion, without axle tubes. Prior to each test the differential housing assembly is completely disassembled and cleaned. After thorough cleaning and coating all internal parts with test oil, the unit is reassembled. The unit is charged with 40 oz of test oil. After driving the unit at 2500 r/min pinion speed, 1 oz of water is added to the oil. Pressure relief system is installed, and when the lubricant temperature reaches 180°F, the motoring phase is continued for four hours at this temperature. The motor is stopped at the completion of the motoring phase, and the test units are removed from the monitoring rig, and placed in a storage box at 125°F for 162 hours. At the end of the storage phase, the test is complete. Differential assembly is drained, disassembled, and rated for rust, stain, and other deposits.

TEST PRECISION

	Intermediate Precision		Reproducibility	
Variable, Merits	$S_{I.P.}$	I.P.	S_R	R
Rust	0.39	1.09	0.44	1.23

Where S = standard deviation.

I.P. = intermediate precision.

R = reproducibility

Information on bias of this test method is not available at present.

MOISTURE OF GREEN PETROLEUM COKE: D 4931

EXPLANATION

Moisture adds weight to coke and serves no useful purpose. Acknowledgement of moisture is thus important in the purchase and sale of green petroleum coke. This test method covers both the preparation procedure for samples containing free water, and the determination of the gross moisture content of green petroleum coke.

TEST SUMMARY

There are two procedures used. *Preparation Procedure* is used when the petroleum coke sample contains free water. The sample is weighed and air dried to equilibrate it with the atmosphere. Determination of the residual moisture is then determined using the *Drying Oven Method*. Air drying and residual moisture are combined to report gross moisture.

The *Drying Oven Method* is used in routine commercial practice when the sample does not contain free water. The sample is crushed to at least –25 mm(1 in.) top sieve size and divided into aliquots of at least 500 g each.

TEST PRECISION

Repeatability:	0.34 %
Reproducibility:	0.58 %

The bias of this test method is not known.

METHYL TERT-BUTYL ETHER BY GAS CHROMATOGRAPHY: D 5441

EXPLANATION

The presence of impurities in methyl tert-butyl ether (MTBE) can have a deleterious effect upon the value of MTBE as a gasoline additive. Oxygenates and olefins if present are of primary concern. This test method provides for the determination of purity of MTBE by gas chromatography. Impurities are determined to a minimum concentration of 0.02 mass %. This test method is not applicable to the determination of MTBE itself in gasoline. Water cannot be measured by this test method, and must be determined by another method and the result used to normalize the chromatographic values. A majority of the impurities in MTBE is resolved by this test method; however, some co-elution is encountered. Cyclopentane and 2,3-dimethylbutane co-elute with MTBE. However, these are not commonly found impurities in MTBE. This test method is inappropriate for impurities that boil at temperatures higher than 180 °C or for impurities that cause poor or no response in a flame ionization detector, such as water.

TEST SUMMARY

Open tubular column gas chromatography with a flame ionization detector used in this test method is a technique that is sensitive to the contaminants commonly found in MTBE, and a technique that is widely used. Each eluting component is identified by comparing its retention time to those established by analyzing standards under identical conditions. The concentration of each component in mass percent is determined by normalization of the peak areas after each peak area is corrected by a detector response multiplication factor. The latter are determined by analyzing prepared standards with concentrations similar to those found in the sample.

TEST PRECISION[1]

The precision of any individual measurement by this test method is expected to be dependent upon several factors such as component volatility, its concentration, and the degree of resolution from other closely eluting components. The table below lists the precision for selected representative components.

Based on the study of pure compounds, this test method was found not to have any significant bias.

Repeatability and Reproducibility for Selected MTBE Components.

Component	Range Weight %	Repeatability	Reproducibility
Methanol	0.0113 to 0.3719	$0.0181^{*}X^{0.25}$	$0.0994^{*}X^{0.25}$
Isobutylene/ 1-butene	0.0168 to 0.1356	$0.0998\ (X+0.0049)$	$0.3199\ (X+0.0049)$
Isopentane	0.0561 to 1.9290	$0.0390^{*}X^{0.6667}$	$0.1646^{*}X^{0.6667}$
Trans-2-pentene	0.0128 to 0.5003	$0.0084^{*}X^{-0.0036}$	$0.0630^{*}X^{0.2678}$
Tert-butanol	0.4741 to 0.8763	0.016	0.132
Cis-2-pentene	0.0970 to 0.5089	$0.0401^{*}X^{0.5}$	$0.1092^{*}X^{0.5}$
2-methyl-2-butene	0.0144 to 0.4391	$0.0122^{*}X^{0.0994}$	$0.0799^{*}X^{0.3818}$
Methyl tert-butyl ether	93.23 to 97.87	$0.0448\ (X/100)^{-18}$	$0.2932\ (X/100)^{-18}$
Sec-butyl methyl ether	0.0200 to 0.4821	$0.0065^{*}X^{0.0123}$	$0.1606^{*}X^{0.4424}$
Tert-amyl methyl ether	0.4961 to 0.7072	0.019	0.124
2,4,4-trimethyl 1-pentene	0.0852 to 1.0150	$0.0388\ (X+0.0415)$	$0.2523\ (X+0.0415)$

[1] This table is Table 3 from Test Method D 5441.

METHYL TERT-BUTYL ETHER IN GASOLINE BY GC: D 4815

EXPLANATION

Ethers, alcohols, and other oxygenates can be added to gasoline to increase octane number and to reduce emissions. Type and concentration of various oxygenates are specified and regulated to ensure acceptable commercial gasoline quality. Drivability, vapor pressure, phase separation, exhaust and evaporative emissions are some of the concerns associated with oxygenated fuels. This test method determines ethers and alcohols in gasoline by gas chromatography, and is applicable to both quality control in the production of gasoline and for the determination of deliberate or extraneous oxygenate additions or contamination.

Individual ethers and alcohols are determined in the concentration range from 0.1 to 20.0, and from 0.1 to 12.0 mass %, respectively. This test method specifically excludes the analysis of alcohol-based fuels such as M-85, E-85, MTBE product, ethanol product, and denatured alcohol. Benzene while detected cannot be quantitated by this test method. Use alternate methods such as Test Methods D 3606 or D 4420. Oxygenates in gasoline can be determined by Test Method D 5599 (see later section).

TEST SUMMARY

A gasoline sample is doped with an internal standard such as 1,2-dimethoxyethane, and is injected into a gas chromatograph equipped with two columns and a column switching valve. The eluted components are detected by a flame ionization or a thermal conductivity detector. The detector response proportional to component concentration is recorded; the peak areas are measured; and the concentration of each component is calculated with reference to the internal standard.

TEST PRECISION

Component	Repeatability	Reproducibility
Methanol (MeOH)	$0.09\ (X^{0.59})$	$0.37\ (X^{0.61})$
Ethanol (EtOH)	$0.06\ (X^{0.61})$	$0.23\ (X^{0.57})$
Isopropanol (iPA)	$0.04\ (X^{0.56})$	$0.42\ (X^{0.67})$
tert-Butanol (tBA)	$0.04\ (X^{0.56})$	$0.19\ (X^{0.67})$
n-Propanol (nPA)	$0.003\ (X^{0.57})$	$0.11\ (X^{0.57})$
MTBE	$0.05\ (X^{0.56})$	$0.12\ (X^{0.67})$
sec-Butanol (sBA)	$0.003\ (X^{0.61})$	$0.44\ (X^{0.67})$
DIPE	$0.08\ (X^{0.56})$	$0.42\ (X^{0.67})$
Isobutanol (iBA)	$0.08\ (X^{0.56})$	$0.42\ (X^{0.67})$
ETBE	$0.05\ (X^{0.82})$	$0.36\ (X^{0.76})$
tert-Pentanol (tAA)	$0.04\ (X^{0.61})$	$0.15\ (X^{0.57})$
n-Butanol (nBA)	$0.06\ (X^{0.61})$	$0.22\ (X^{0.57})$
TAME	$0.05\ (X^{0.70})$	$0.31\ (X^{0.51})$
Total Oxygen	$0.02\ (X^{1.26})$	$0.09\ (X^{1.27})$

Where X is the mean mass percent of the component.

METHYL TERT-BUTYL ETHER IN GASOLINE BY INFRARED SPECTROSCOPY:

D 5845

EXPLANATION

See the discussion under Test Method D 4815. This infrared method is faster, simpler, less expensive and more portable than the current methods. It measures MTBE and other alcohols in the concentration ranges from about 0.1 to about 20 mass %. This test method, however, cannot be used for compliance with federal regulations.

Oxygenates in gasoline can be determined using the Test Method D 5599 (see Oxygenates section).

TEST SUMMARY

A sample of gasoline is analyzed by infrared spectroscopy. A multivariate mathematical analysis is carried out which converts the detector response for the selected regions in the spectrum of an unknown to a concentration of each component.

TEST PRECISION

Oxygenate	Repeatability	Reproducibility
MTBE	0.13 mass %	0.98 mass %
TAME	0.13	1.36
ETBE	0.15	0.77
Ethanol	0.13	0.59
Methanol	0.07	0.37
t-Butanol	0.10	0.59
DIPE	0.14	0.79
Total Oxygen Content	0.05	0.30

Biases were observed in the round robin; however, it is not possible to make a definitive statement on this issue.

NAPTHALENE HYDROCARBONS IN AVIATION TURBINE FUELS BY ULTRAVIOLET (UV) SPECTROPHOTOMETRY:

D 1840

EXPLANATION

Napthalenes when burned tend to have a relatively larger contribution to a sooty flame, smoke, and thermal radiation than single ring aromatics. This test method covers the determination of the total concentration of napthalene, acenapthene, and alkylated derivatives of these hydrocarbons in straight-run jet fuels containing no more than 5 % of such components and having end points below 600°F. This test method determines the maximum amount of napthalenes that could be present.

TEST SUMMARY

The total concentration of napthalenes in jet fuels is measured by absorbance at 285 nm of a solution of the fuel at known concentration. A number of compounds interfere. This can be minimized by the end point limitation of 600°F. Saturated hydrocarbons, olefins, thiophenes and alkyl or cycloalkyl derivatives of benzene will not interfere.

TEST PRECISION

Procedure	Range Naphthalenes, vol. %	Repeatability	Reproducibility
A	0.03–4.25	$0.0222(1.00+X)$	$0.0299(1.00+X)$
B	0.08–5.6	$0.056\ X^{0.6}$	$0.094\ X^{0.6}$

Where X is the mean result.

Bias of this test method cannot be determined.

NEEDLE PENETRATION OF PETROLEUM WAXES: D 1321

(Equivalent Test Methods: IP 49, DIN 51579, and AFNOR T66-004)

EXPLANATION

Petroleum waxes differ in hardness. Hardness may have a significant effect upon other physical properties. Needle penetration is a measurement of hardness. This method is applicable to waxes having a penetration of not greater than 250.

This test method is similar to the needle penetration method for determining the penetration of bituminous material (Test Method D 5). Similar cone methods applicable to greases and to petrolatum are described in Test Methods D 217 and D 937, respectively.

TEST SUMMARY

The sample of wax is melted, heated to 17°C(30°F) above its congealing point, poured into a container, and then air cooled under controlled conditions. The sample is then conditioned at test temperature in a water bath. Penetration is measured with a penetrometer, which applies a standard needle to the sample for 5 s under a load of 100 g.

TEST PRECISION

Repeatability:	$1.72\ (10^{0.00524X})$
Reproducibility:	$4.81\ (10^{0.00442X})$

Where X is penetration.

This procedure has no bias.

NITROGEN DETERMINATION TRACE NITROGEN BY OXIDATIVE COMBUSTION AND CHEMILUMINESCENCE DETECTION: D 4629

GENERAL

The concentration of nitrogen is a measure of the presence of nitrogen-containing additives. Knowledge of its concentration can be used to predict performance.

There are two ways of determining nitrogen in additives: Kjeldahl method and instrumental combustion method. A comparison of these methods is given in Table 6.

EXPLANATION

This test method covers the determination of the trace nitrogen naturally found in liquid hydrocarbons boiling in the range from approximately 50 to 400°C, with viscosities between approximately 0.2 and 10 cSt(mm^2/s) at room temperature. This test method is applicable to naphthas, distillates, and oils containing 0.3 to 100 mg/kg total nitrogen.

TEST SUMMARY

The sample of liquid petroleum hydrocarbon is injected into a stream of inert gas (helium or argon). The sample is vaporized and carried to a high temperature zone where oxygen is introduced and organic and bound nitrogen is converted to nitric oxide (NO). The NO contacts ozone and is converted to excited nitrogen oxide (NO_2). The light emitted as the excited NO_2 decays is detected by a photomultiplier tube and the resulting signal is a measure of the nitrogen contained in the sample.

TEST PRECISION

Reproducibility:	$0.15\ (X)^{0.54}$
Repeatability:	$0.85\ (X)^{0.54}$

Where X is the average of two test results.

Comparison of Nitrogen Determination Test Methods.

Test Method	D 3228	D 4629	D 5291	D 5762
Technique	Kjeldahl: acid digestion and titration	Syringe injection, combustion, and chemiluminescence detection	Combustion, separation, and specific detection. Three procedures	Boat inlet injection, combustion, and chemiluminescence detection
Scope	Lube oils, fuel oils, additives	Liquid hydrocarbons, napthas, oils, distillates	Crude oil, fuel oils, lube oils, additives	Lube oils, liquid hydrocarbons
N Range	0.03–0.1 % in lube oils; 0.015–2.0 % in fuel oils	0.3–100 mg/kg	0.75–2.5 %	40–10 000 mg/kg
Limitations	Not applicable to N—O or N—N bonds		Not applicable to gasolines or aviation tubine fuels	
Sample Size	1–1.5 g	3–40 μL	2–200 mg	<10 mg
Repeatability	Lube oils: 0.01 % Fuel oils: $0.066\sqrt{M}$	$0.15X^{0.54}$	0.1670	$0.142X^{0.92}$ mg/kg
Reproducibility	Lube oils: 0.02 % Fuel oils: $0.19\sqrt{X}$	$0.85X^{0.54}$	0.4456	$0.371X^{0.92}$ mg/kg
Bias	Not known	Not known	Not known	None

NITROGEN DETERMINATION BY BOAT-INLET CHEMILUMINESCENCE:

D 5762

EXPLANATION

This test method covers the determination of nitrogen in liquid hydrocarbons including petroleum process streams and lubricating oils in the concentration range from 40 to 10 000 μg/g nitrogen. For light hydrocarbons containing less than 100 μg/g nitrogen, Test Method D 4629 can be more appropriate.

TEST SUMMARY

A hydrocarbon sample is placed on a sample boat at room temperature. The sample and boat are advanced into a high temperature combustion tube where the nitrogen is oxidized to nitric oxide (NO) in an oxygen atmosphere. The NO contacts ozone, is converted to excited nitrogen dioxide (NO_2). The light emitted as the excited NO_2 decays is detected by a photomultiplier tube and the resulting signal is a measure of the nitrogen contained in the sample.

TEST PRECISION

Repeatability:	0.099 *X*
Reproducibility:	0.291 *X*

Where *X* is the average of results obtained.

NITROGEN DETERMINATION OF CARBON, HYDROGEN, AND NITROGEN:

D 5291

EXPLANATION

This is the only ASTM method for simultaneous determination of carbon, hydrogen, and nitrogen in petroleum products and lubricants. There are at least three instrumental techniques available for this analysis, each based on different chemical principles. However, all involve sample combustion, components separation, and final detection. These test methods are applicable to lubricating oils, crude oils, additives, and residues. These test methods were tested in the concentration range of at least 75 to 87 mass % for carbon, at least 9 to 16 mass % for hydrogen, and 0.1 to 2 mass % for nitrogen. The nitrogen method is not applicable to light materials or to samples containing <0.75 mass % nitrogen, or both, such as gasoline, jet fuel, naptha, diesel fuel, or chemical solvents. These test methods are not recommended for the analysis of volatile materials such as gasoline, gasoline oxygenate blends, or aviation turbine fuels.

TEST SUMMARY

In *Test Method A* (*Leco Instrument*), a sample is combusted in an oxygen atmosphere, and the product gases are separated from each other by adsorption over chemical agents. The remaining elemental nitrogen gas is measured by a thermal conductivity cell. Carbon and hydrogen are separately measured by selective infrared cells as carbon dioxide and water.

In *Test Method B* (*Perkin Elmer Instrument*), a sample is combusted in an oxygen atmosphere, and the product gases are separated from each other and the three gases of interest are measured by gas chromatography.

In *Test Method C* (*Carlo Erba Instrument*), a sample is combusted in an oxygen atmosphere, and the product gases are cleaned by passage over chemical agents. The three gases of interest are chromatographically separated and measured with a thermal conductivity detector.

TEST PRECISION

Element	Range, Mass %	Repeatability	Reproducibility
Carbon	75–87	$(x+48.48)0.0072$	$(X+48.48)0.018$
Hydrogen	9–19	$(x^{0.5})0.1162$	$(X^{0.5})0.2314$
Nitrogen	0.75–2.5	0.1670	0.4456

This test method has no relative bias among three procedures.

NITROGEN DETERMINATION KJELDAHL METHOD:

D 3228

EXPLANATION

This test method is applicable for the determination of nitrogen in lubricating oils when present in the concentration from 0.03 to 0.10 weight %, and for the determination of nitrogen in fuel oils when present in the concentration from 0.015 to 2.0 weight %. This test method may not be applicable to certain materials containing N—O, N—N linkage, and thus in such materials may produce low nitrogen results compared to other instrumental methods, such as Test Methods D 4629, D 5291, or D 5762.

TEST SUMMARY

The sample is digested in a mixture of concentrated sulfuric acid, potassium sulfate, mercuric oxide, and copper sulfate. After digestion, sodium sulfide is added to precipitate the mercury, and the mixture is made alkaline with caustic. Nitrogen, now in the form of ammonia, is distilled into a boric acid solution. The ammonia is titrated with standard sulfuric acid using methyl purple as an indicator.

TEST PRECISION

Sample	Repeatability	Reproducibility
Lubricating Oils	0.01 wt. %	0.02 wt. %
Fuel Oils	$0.066\sqrt{M}$	$0.19\sqrt{M}$

Where M = mean of two values.

ODOR OF PETROLEUM WAX:
D 1833

EXPLANATION

Odor intensity of wax is an important property in some uses of wax such as food packaging. It is often included in the specifications of petroleum wax. This method gives a basis for agreement between laboratories on the odor intensity of wax using a numerical scale rather than descriptive terms. The rating results, however, can be influenced by odor type.

TEST SUMMARY

About 10 g of thin shavings from a petroleum wax sample are placed on odor-free paper or glassine. They are then evaluated by each panel member and assigned a number corresponding to a number of the odor scale description best fitting the intensity of the odor. Alternatively, the wax shavings are placed in bottles, and each panel member makes the evaluation between 15 and 60 min after the specimens are prepared. A panel should have at least five people. The average of the panel rating is reported as the odor rating of the sample.

TEST PRECISION

This test method has no bias. However, precision data have not been developed.

AUTOMOTIVE ENGINE OIL COMPATABILITY WITH TYPICAL SEAL ELASTOMERS:
D 7216

EXPLANATION

Some engine oil formulations have been shown to lack compatibility with certain elastomers used for seals in automotive engines. These deleterious effects on the elastomer are greatest with new engine oils and when the exposure is at elevated temperatures. This test method covers quantitative procedures for the evaluation of the compatibility of automotive engine oils with four reference elastomers typical of those used in the sealing materials in contact with these oils. Compatibility is evaluated by determining the changes in volume, Durometer A hardness and tensile properties when the elastomer specimens are immersed in the oil for a specified time and temperature. Effective sealing action requires that the physical properties of elastomers used for any seal have a high level of resistance to the liquid or oil in which they are immersed. When such a high level of resistance exists, the elastomer is said to be compatible with the liquid or oil.

This test method provides a preliminary or first order evaluation of oil/elastometer compatibility only. Because seals may be subjected to static or dynamic loads, or both, and they may operate over a range of conditions, a complete evaluation of the potential sealing performance of any elastomer/oil combination in any service condition usually requires tests additional to those described in this test method. The four reference elastomer formulations specified in this test method were chosen to be representative of those used in heavy-duty diesel engines. The procedures described in this test method can, however, be used to evaluate the compatibility of automotive engine oils with different elastomer types/formulations or different durations and temperatures to those employed in this test method.

This test method requires that non-reference oil(s) be tested in parallel with a reference oil, TMC 1006, known to be aggressive for some parameters under service conditions. This relative compatibility permits decisions on the anticipated or predicted performance of the non-reference oil in service. Elastomer materials can show significant variation in physical properties, not only from batch-to-batch but also within a sheet and from sheet-to-sheet. Results obtained with the reference oil are submitted by the test laboratories to the TMC to allow it to update continually the total and within-laboratory standard deviation estimates. These estimates, therefore, incorporate effects of variations in the properties of the reference elastomers on the test variability.

TEST SUMMARY

Measurements of initial volume, hardness (Durometer A) and tensile properties (ultimate elongation and tensile strength) are made on specimens of specified dimensions cut from sheets of reference elastomers. These elastomer specimens are immersed in both non-reference oil(s) and a reference oil and aged for 336 h at specified temperatures. The effects of the test oil on the elastomers are determined by measuring the changes in volume, hardness, and tensile properties resulting from the immersion in the oil.

TEST PRECISION

See table—for precision obtainable by this test method. No estimate of the bias for this procedure is possible because the performance results for an oil are determined only under the specific conditions of the test and no absolute standard exists.

Reference Oil Precision Statistics[a]

Elastomer[b]	Precision Parameter	% Volume Change	Durometer A Hardness Change, points	% Tensile Strength Change	% Ultimate Elongation Change
Nitrile (NBR)	i.p.	2.7	4.0	20.4	20.1
Nitrile (NBR)	R	2.7	5.1	21.3	20.1
Silicone (VMQ)	i.p.	6.8	4.5	15.0	27.1
Silicone (VMQ)	R	6.8	7.4	15.0	27.1
Polyacrylate (ACM)	i.p.	2.3	5.4	23.6	26.6
Polyacrylate (ACM)	R	2.4	5.5	23.7	27.1
Fluoroelastomer (FKM)	i.p.	0.36	5.2	14.8	22.9
Fluoroelastomer (FKM)	R	0.45	6.8	15.6	29.3

[a] These statistics were obtained from the TMC (ftp://ftp.astmtmc.cmu.edu) and were applicable on Oct. 16, 2003. They are based on data on Reference Oil TMC 1006 provided by five laboratories between Nov. 1, 2001 and Oct. 16, 2003 and involve approximately 600 individual data points for each elastomer/property combination. All laboratories used an earlier draft of this test method.

[b] See Table 1 for full identification of the elastomer types.

OIL CONTENT OF PETROLEUM WAXES:
D 721

EXPLANATION

The oil content of a wax may have significant effects on several of its properties such as strength, hardness, flexibility, scuff resistance, coefficient of friction, coefficient of expansion, melting point, and oil straining. The importance of these effects may be dependent upon the ultimate use of the wax. This test method determines the oil content of the petroleum waxes with a congealing point of 30°C(86°F) or higher as determined by the Test Method D 938, and containing no more than 15 % of oil. With some types of waxes of oil content greater than 5 %, there may be incompatibility with methyl ethyl keytone (MEK) resulting in the formation of two liquid phases. In such cases this test method cannot be used.

TEST SUMMARY

A wax sample is dissolved in methyl ethyl ketone (MEK), the solution cooled to −32°C(−25°F) to precipitate the wax, and filtered. The oil content of the filtrate is determined by evaporating the MEK solvent and weighing the residue.

TEST PRECISION

Repeatability:	0.06+8 % of the mean
Reproducibility:	0.2+11 % of the mean

This test method has no bias.

OIL SEPARATION FROM GREASE BY CENTRIFUGING (KOPPERS METHOD):
D 4425

EXPLANATION

This test method is useful in evaluating the degree to which a grease would separate into fluid and solid components when subjected to high centrifugal forces. Flexible shaft couplings, universal joints, and rolling element thrust bearings are examples of machinery which subject lubricating greases to large and prolonged centrifugal forces. This test method has been found to give results that correlate well with results from actual service. The test method may be run at other conditions with agreement between parties but the precision given in this test method may not apply.

TEST SUMMARY

Pairs of centrifuge tubes are charged with grease samples and are placed in the centrifuge. The grease samples are subjected to a centrifugal force equivalent to a G value of 36 000 at 50°C ± 1°C, for specific periods of time. The resistance of the grease to separate the oil is then defined as a ratio of the percent oil separated to the total number of hours of testing.

TEST PRECISION

Repeatability:	4.8 % points of value of oil separation in vol. %
Reproducibility:	18.7 % points of value of oil separation in vol. %

The procedure in this test method has no bias because the value can be defined only in terms of the test method.

OIL SEPARATION FROM GREASE (CONICAL SIEVE METHOD):
D 6184

EXPLANATION

When a lubricating grease separates oil, the residual material changes in consistency which can affect the ability of the product to function as designed. Test results obtained with this procedure are intended to correlate with oil separation that occurs in 35 lb pails of grease during storage. They are not untended to predict oil separation tendencies of grease under dynamic service conditions. No quantitative relationship between results and oil separation in 35 lb pails have been developed. No data have been developed to compare results between this test method and Test Method D 1742 Oil Separation from Greases During Storage.

The conical sieve method for measuring leakage from lubricating grease (commonly known as the cone bleed test) is virtually the same as Federal Test Method 791C Method 321.3. The test method is conducted at 100°C for 30 h unless other conditions are required by the grease specification. This test method is not suitable for greases having a penetration (Test Method D 217) greater than 340 (softer than NLGI No. 1 Grade).

TEST SUMMARY

A weighed sample is placed in a cone shaped, wire-cloth sieve, suspended in a beaker, then heated under static conditions for the specified time and temperature. Unless otherwise required by the grease specification, the sample is tested at standard conditions of 100 ± 0.5°C for 30 ± 0.25 h. At the end the separated oil is weighed and reported as a percentage of the mass of the starting test sample.

TEST PRECISION

Repeatability:	$1.151 \times (M)^{0.5}$
Reproducibility:	$1.517 \times (M)^{0.5}$

Where M is the mean of two determinations. The values above are expressed as % oil separation.

This method has no bias because the value of the oil loss is defined only in terms of this test method.

OLEFINS IN ENGINE FUELS BY GC:
D 6296

EXPLANATION

The quantitative determination of olefins in spark ignition engine fuels is required by government regulations. Knowledge of the total olefin content provides a means to monitor the efficiency of catalytic cracking processes. This test method can determine olefins in the C_4 to C_{10} range in spark ignition engine fuels or related hydrocarbon streams such as napthas and cracked napthas. Olefin concentrations in the range 0.2 to 5.0 volume % or mass % or both can be determined. Samples with higher olefins content can also be analyzed after proper dilutions. This test method is applicable to samples containing alcohols and ethers; however, samples containing greater than 15 % alcohol must be diluted. Samples containing greater than 5 % ether must also be diluted to 5 % or less level. When ethyl-ter-butyl ether is present, only olefins in the C_4 to C_9 range can be determined. The test method is not applicable to M85 or E85 fuels, which contain 85 % methanol and ethanol, respectively.

This test method provides better precision for olefin analysis than Test Method D 1319. It is also much faster. Other alternate methods for olefin determination are Test Method D 1319 which is the EPA mandated method, and Test Method D 6293, which is the alternate method used for the California Air Resources Board (CARB) for this analysis.

TEST SUMMARY

A sample of fuel is injected into a computer controlled gas chromatographic system which consists of a series of columns, traps, and switching valves operating at various temperatures. The final eluted olefins are detected by flame ionization detector. Quantitation is provided by comparing the olefins' peak areas with that of an external standard. Some types of sulfur compounds may interfere by reducing the trap capacity for olefins. Commercial dyes and additives added to the fuels, and dissolved water have not been found to interfere. Free water, however, must be removed before the analysis.

TEST PRECISION

Repeatability:	$0.074\ X^{0.72}$
Reproducibility:	$0.26\ X^{0.72}$

Where X is the average of two results in volume %.

Bias of this test method is not known.

OLEFINS IN GASOLINES BY SUPERCRITICAL FLUID CHROMATOGRAPHY:

D 6550

EXPLANATION

Gasoline-range olefinic hydrocarbons contribute to photochemical reactions in the atmosphere, which result in the formation of photochemical smog in susceptible urban areas. The California Air Resources Board (CARB) has specified a maximum allowable limit of total olefins in motor gasoline. This test method compares favorably with Test Method D 1319 (FIA) for the determination of total olefins in motor gasolines. It does not require any sample preparation, has a comparatively short analysis time of about 10 min, and is readily automated. The application range is 1–25 mass % of total olefins. This test method can be used for analysis of commercial gasolines, including those containing varying levels of oxygenates, such as methyl tert/butyl ether (MTBE), diisopropyl ether (DIPE), methyl tert/amyl ether (TAME), and ethanol, without interference. This method has not been designed for the determination of the total amounts of saturates, aromatics, and oxygenates. Alternative methods for determination of olefins in gasoline include Test Methods D 6293 and D 6296.

TEST SUMMARY

A small aliquot of the fuel sample is injected onto a set of two chromatographic columns connected in series and transported using supercritical carbon dioxide as the mobile phase. The first column is packed with high-surface-area silica particles. The second column contains either high-surface-area silica particles coated with silver ions or strong-cation-exchange material loaded with silver ions. Two switching valves are used to direct the different classes of components through the chromatographic system to the detector. In a forward flow mode, saturates (normal and branched alkanes, cyclic alkanes) pass through both columns to the detector, while the olefins are trapped on the silver-loaded column, and the aromatics and oxygenates are retained on the silica column. The latter two are subsequently eluted from the silica column to the detector in a back-flush mode. Finally, the olefins are back-flushed from the silver-coated column to the detector. A flame ionization detector (FID) is used for quantitation. Calibration is based on the area of the chromatographic signal for olefins, relative to standard reference materials, which contain a known mass % of total olefins as corrected for density.

TEST PRECISION

Repeatability:	$0.11\ X^{0.75}$
Reproducibility:	$0.47\ X^{0.75}$

Where X is the mean concentration between 1 and 25 mass %.

Since there is no material with an accepted reference value available, no statement regarding bias of this method can be made.

OXIDATION INDUCTION TIME OF GREASES: D 5483

EXPLANATION

Oxidation induction time can be used as an indication of oxidation stability. No correlation has been determined between the results of this test and service performance. This test method covers lubricating greases subjected to oxygen at 3.5 MPa(500 psig) and temperatures between 155 and 210°C.

TEST SUMMARY

A small quantity of grease in a sample pan is placed in a test cell. The cell is heated to a specified temperature and then pressurized with oxygen. The cell is held at a regulated temperature and pressure until an exothermic reaction occurs. The extrapolated onset time is measured and reported as the oxidation induction time. A kinetic equation incorporated with this test method can estimate oxidation induction times at other temperatures.

TEST PRECISION

Determinability:	$0.59\sqrt{M}$
Repeatability:	$0.42\sqrt{M}$
Reproducibility:	$0.71\sqrt{M}$

Where M is the mean of the two results.

This procedure has no bias.

OXIDATION INDUCTION TIME OF LUBE OILS BY PRESSURE DIFFERENTIAL SCANNING CALORIMETRY (PDSC):

D 6186

EXPLANATION

Oxidation induction time as determined under the conditions of this test method, may be used as an indication of oxidation stability. This test method covers the determination of oxidation induction time of lubricating oils subjected to oxygen at 3.5 MPa and temperature between 130 and 210°C. This test method is faster than other oil oxidation tests, and requires a very small amount of sample. No correlation has been established between the results of this test method and service performance.

TEST SUMMARY

A small quantity of oil is weighed into a sample pan and placed in a test cell. The cell is heated to a specified temperature and then pressurized with oxygen. The cell is held at a regulated temperature and pressure until an exothermic reaction occurs. The extrapolated onset time is measured and reported as the oxidation induction time for the lubricating oil at the specified test temperature.

TEST PRECISION

Repeatability	0.17 m
Reproducibility	0.35 m

Where m is the average of two test results.

The procedure in this test method has no bias because the value of oxidation induction time can be defined only in terms of this test method.

OXIDATION OF USED LUBRICANTS BY FT-IR USING PEAK AREA INCREASE CALCULATION:

D 7214

EXPLANATION

The peak area increase (PAI) is representative of the quantity of all the compounds containing a carbonyl function that have formed by the oxidation of the lubricant (aldehydes, ketones, carboxylic acids, esters, anhydrides, etc.). The PAI gives representative information on the chemical degradation of the lubricant which has been caused by oxidation. This test was developed for transmission oils and is used in the CEC-L-48-A-00 test as a parameter for the end of test evaluation. However, the test method is not intended to measure an absolute oxidation property that can be used to predict performance of an oil in service. The test may be used for other than in-service transmission oils also; however, the stated precision may not apply.

TEST SUMMARY

FT-IR spectra of the fresh and the used oils are recorded in a transmission cell of known pathlength. Both spectra are converted to absorbance mode and then subtracted. Using this resulting differential spectrum, a baseline is set under the peak corresponding to the carbonyl region around 1650 cm^{-1} and 1820 cm^{-1} and the area created by this baseline and the carbonyl peak is calculated. The area of the carbonyl region is divided by the cell pathlength in mm and this result is reported as peak area increase.

INTERFERENCES—Some specific cases (very viscous oil, use of ester as base stock, high soot content) may require dilution of the sample and a specific area calculation as described in the method. The results of this test method may be affected by the presence of other components with an absorbance band in the zone of 1600 to 1800 cm^{-1}. Low PAI values may be difficult to determine in those cases.

TEST PRECISION

Repeatability:	$0.029(x+9)$
Reproducibility:	$0.26(x+35)$

Where x is the average of two results.

The bias of this test method cannot be determined because there is no certified reference standard for these properties available.

OXIDATION STABILITY OF AVIATION FUELS:

D 873

(Equivalent Test Methods: IP 138 and AFNOR M07-013)

EXPLANATION

This test method covers the determination of the tendency of the aviation reciprocating, turbine, and jet engine fuels to form gum and deposits under accelerated aging conditions. The tendency of the fuels to form gum and deposits in these tests has not been correlated with field performance (and can vary markedly) with the formation of gum and deposits under different storage conditions. This test method is not intended for determining the stability of fuel components, particularly those with a high percentage of low boiling unsaturated compounds, as they may cause explosive conditions within the apparatus.

TEST SUMMARY

The fuel is oxidized under prescribed conditions in a pressure vessel filled with oxygen. The amounts of soluble gum, insoluble gum, and precipitates are weighed.

TEST PRECISION

	Repeatability		Reproducibility	
Analysis	Reciprocating Engine Fuel	Turbine Fuel	Reciprocating Engine Fuel	Turbine Fuel
Potential Gum mg/100 mL Up to 5	2	2	3	4
Over 5-10	3	3	4	5
Over 10-20	4	5	6	7
Precipitate mg/100 mL Up to 2	1		1	

The bias of this test method has not been determined.

OXIDATION STABILITY OF DISTILLATE FUELS:
D 2274
(Equivalent Test Methods: IP 388 and ISO 12205)

EXPLANATION

This test method provides a basis for the estimation of the storage stability of middle distillate fuels with an IBP above 175°C and a 90 % recovery point below 370°C such as No. 2 fuel oil. It is not applicable to fuels containing residual oil, or any significant component derived from a nonpetroleum source. This test method may not provide a prediction of the quantity of insolubles that will form in field over any given period of time. This test method yields results more rapidly than Test Method D 2274; however, the results by these two test methods may not be similar.

TEST SUMMARY

A 350-mL sample of filtered middle distillate fuel is aged at 95°C(203°F) for 16 h while oxygen is bubbled through the sample at a rate of 3 L/h. Next the sample is cooled to approximately room temperature before filtering to obtain the filterable insoluble quantity. Adherent insolubles are then removed from the oxidation cell and associated glassware with trisolvent. The trisolvent is evaporated to obtain the quantity of adherent insolubles. The sum of these two expressed as mg/100 mL is reported as total insolubles.

TEST PRECISION

Repeatability: 0.54(total insolubles)$^{-4}$
Reproducibility: 1.06(total insolubles)$^{-4}$

The bias of this test method is not known.

OXIDATION STABILITY OF OILS BY THIN FILM OXYGEN UPTAKE (TFOUT):
D 4742

EXPLANATION

This test method is used to evaluate oxidation stability of lubricating base oils with additives in the presence of chemistries similar to those found in gasoline engine service. Test results for some ASTM reference oils have been found to correlate with sequence IIID engine test results; however, this test is not a substitute for the engine testing of an engine oil in established engine tests. This test method can be used for engine oils with viscosity in the range from 4 cSt to 21 cSt at 100°C including the re-refined oils.

TEST SUMMARY

The oil is mixed in a glass container with three other liquids that are used to simulate engine conditions: an oxidized/nitrated fuel component, a mixture of soluble metal naphthenates (lead, copper, iron, manganese, and silicon), and distilled water. The container with the oil mixture is placed in a high pressure reactor equipped with a pressure gage. The reactor is sealed and charged with oxygen to a pressure of 620 kPa, and placed in an oil bath at 160°C at an angle of 30° from the horizontal. The reactor is rotated axially at a speed of 100 r/min forming a thin film of oil within the glass container resulting in a relatively large oil-oxygen contact area. The pressure of the reactor is continuously recorded from the beginning of the test. The test is terminated when a rapid decrease of the reactor pressure is observed. The time period between these two events is called the oxidation induction time and is used as a measure of the relative oil oxidation stability.

TEST PRECISION

Repeatability:	0.10 (X+5min)
Reproducibility:	0.22 (X+5min)

Where X is the mean of the results.

A bias statement is now being developed.

OXIDATION STABILITY OF EXTREME PRESSURE LUBRICATING OILS:
D 2893

EXPLANATION

This test method has been widely used to measure the oxidation stability of extreme pressure lubricating fluids, gear oils, and mineral oils. The changes in the lubricant resulting from this test method are not always associated with oxidation of the lubricant. Some changes may be due to thermal degradation. Many of the new extreme pressure oils cannot reasonably be evaluated by this test method.

TEST SUMMARY

The oil sample is heated to a temperature of 95°C in the presence of dry air for 312 h. The oil is then tested for precipitation number (by Test Method D 91), and increased in kinematic viscosity (by Test Method D 445).

TEST PRECISION

This test method has no bias.

See the repeatability and reproducibility in Figs. 11 and 12.

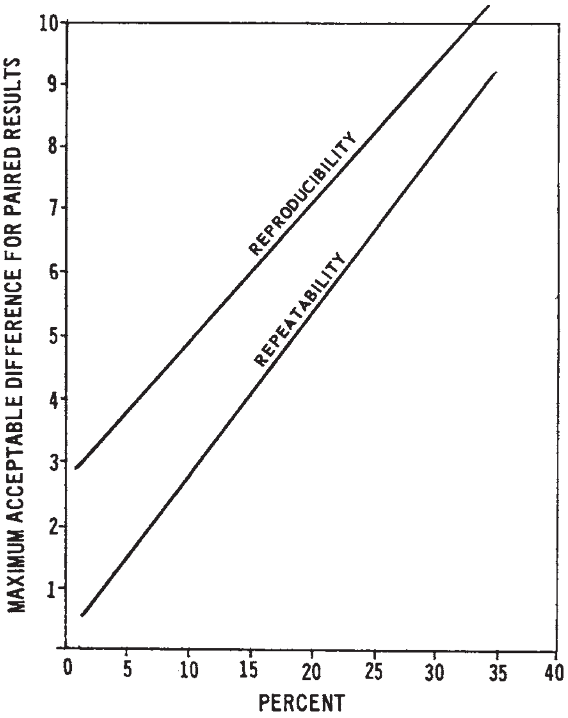

FIG. 11—*Precision Data Viscosity Increase.*

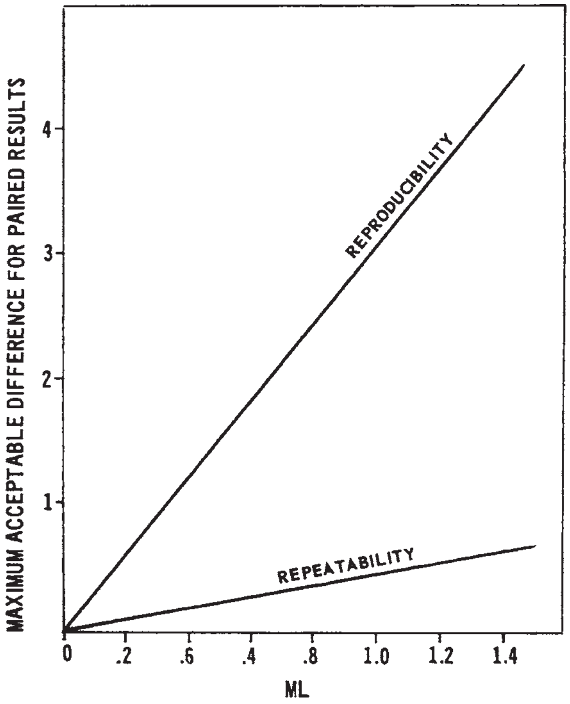

FIG. 12—*Precision Data, Precipitation Number Increase.*

OXIDATION STABILITY OF GASOLINE: D 525 (Equivalent Test Methods: IP 40, ISO 7536, DIN 51780, and AFNOR M07-012)

EXPLANATION

The induction period may be used as an indication of the tendency of motor gasoline to form gum in storage. However, its correlation with the formation of gum in storage may vary markedly under different storage conditions and with different gasolines.

This test method is not intended for determining the stability of gasoline components, particularly those with a high percentage of low boiling unsaturated compounds, as they may cause explosive conditions within the apparatus.

TEST SUMMARY

The sample is oxidized in a pressure vessel initially filled at 15 to 25°C with oxygen pressure at 680 to 703 kPa and heated at a temperature between 98 and 102°C. The pressure is recorded continuously at stated intervals until the breakpoint is reached. The time required for the sample to reach this point is the observed induction period at the temperature of the test from which the induction period at 100°C can be calculated.

TEST PRECISION

Repeatability:	5 %
Reproducibility:	10 %

The bias for this test method has not been determined.

OXIDATION STABILITY OF GEAR OILS: D 5763

EXPLANATION

Degradation of gear oils by oxidation or thermal breakdown, or both, can result in sludge buildup and render the oil unsuitable for further use as a lubricant. This test method covers the determination of the oxidation characteristics of extreme pressure and nonextreme pressure gear oils, and includes the quantitative determination of total sludge, viscosity change, and oil loss. However, the test results may or may not correlate with the field service performance of the gear oils. This test method is a modification of Test Method D 2893 (see previously), which measures the viscosity increase and precipitation number of the oil stressed at 95°C, but does not measure the amount of the sludge formed.

TEST SUMMARY

The oil sample is heated to a temperature of 120°C for 312 h while dry air is passed through it at a rate of 3 L/h. At the end the sample is cooled to room temperature and the apparatus is reweighed to determine the loss. Sludge is filtered through a vacuum filter. Viscosity of the filter oil is determined by Test Method D 445.

TEST PRECISION

Analysis	Repeatability	Reproducibility
Total Sludge	0.42 X, g %	1.4 X, g %
Viscosity Increase	$0.058X^2$, %	$0.14X^2$, %

Where X is the found mean value.

The precision of oil loss has not been determined.

The bias of this test method has not been determined.

OXIDATION STABILITY OF INHIBITED MINERAL OILS: D 943

(Equivalent Test Method: ISO 4263)

EXPLANATION

This test method is widely used for specification purposes and is considered of value when estimating the oxidation stability of lubricants, especially those that are prone to water contamination. However, the correlation between these results and the oxidation stability of a lubricant in field service may vary markedly. This test method evaluates the oxidation stability of inhibited steam-turbine oils in the presence of oxygen, water, and copper and iron metals at elevated temperature. This test method is also used for testing other oils such as hydraulic and circulating oils having a specific gravity less than that of water and containing rust and oxidation inhibitors.

TEST SUMMARY

The oil sample is contacted with oxygen in the presence of water and an iron-copper catalyst at 95°C. The test continues until the measured total acid number of the oil is 2.0 mg KOH/g or above. The number of test hours required for the oil to reach this value is called the oxidation lifetime.

TEST PRECISION

Repeatability:	0.192 X
Reproducibility:	0.332 X

Where X is the mean value.

OXIDATION STABILITY OF LUBRICATING GREASES: D 942

(Equivalent Test Methods: IP 142 and DIN 51808)

EXPLANATION

This test method measures the net change in pressure resulting from oxygen consumption by oxidation and gain in pressure due to the formation of volatile oxidation byproducts. This test method does not predict the stability of greases under dynamic service conditions, nor the stability of greases stored in containers for long periods, nor the stability of films of greases on bearings and motor parts. It should not be used to estimate the relative oxidation resistance of different grease types.

TEST SUMMARY

The sample of grease is oxidized in a bomb heated to 99°C and filled with oxygen at 110 psi. Pressure is observed and recorded at stated intervals. The degree of oxidation after a given period of time is determined by the corresponding decrease in oxygen pressure.

TEST PRECISION

Mean Pressure Drop, psi	Repeatability, psi	Reproducibility, psi
0 to 5	2	3
Over 5 to 10	3	5
Over 10 to 20	6	8
Over 20 to 55	10	20

This procedure has no bias.

OXIDATION STABILITY OF OILS BY UNIVERSAL OXIDATION TEST:
D 5846

EXPLANATION

Degradation of hydraulic fluids and turbine oils, because of oxidation or thermal breakdown, can result in the formation of acids or insoluble solids and render the oil unfit for further use. This test method covers a procedure for evaluating the oxidation stability of such oils. Correlation between the results of this test method and the oxidation stability in use can vary markedly with service conditions and with various oils.

TEST SUMMARY

An oil sample is contacted with air at 135°C in the presence of copper and iron metals. The acid number and spot forming tendency of the oil are measured daily. The test is terminated when the oxidation life of the oil has been reached. The oil is considered degraded when either its acid number (measured by Test Methods D 664 or D 974) has increased by 0.5 mg KOH/g over that of new oil; or when the oil begins to form insoluble solids as evident from a clearly defined dark spot surrounded by a ring of clear oil when a drop of the oil is placed on a filter paper.

TEST PRECISION

Samples	Repeatability	Reproducibility
Antiwear Hydraulic Oils	0.0614 X	0.0918 X
Steam and Gas Turbine Oils	0.0486 X	0.1400 X

Where X is the mean value of the oxidation test life.

This test method has no bias.

OXIDATION STABILITY OF STEAM TURBINE OILS BY ROTATING PRESSURE VESSEL:
D 2272

EXPLANATION

The estimate of oxidation stability is useful in controlling the continuity of this property for batch acceptance of production lots having the same operation. This test method utilizes an oxygen pressured vessel to evaluate the oxidation stability of new and in-service turbine oils having the same composition (base stock and additives) in the presence of water and a copper catalyst coil at 150°C. It is not intended that this test method be a substitute for Test Method D 943 or be used to compare the service lives of new oils of different compositions. This test method is also used to assess the remaining oxidation test life of in-service oils. A modification of the rotating vessel method has been published as Test Method D 2112, which uses a similar procedure and apparatus but a lower (140°C) bath temperature. That method requires duplicate testing; Test Method D 2272 also conducted duplicate testing in the past.

TEST SUMMARY

The test oil, water, and copper catalyst coil, contained in a covered glass container, are placed in a vessel equipped with a pressure gage. The vessel is charged with oxygen to a gage pressure of 620 kPa, placed in a constant temperature oil bath set at 150°C, and rotated axially at 100 r/min at an angle of 30° from the horizontal. The number of minutes required to reach a specific drop in gage pressure is the oxidation stability of the test sample.

TEST PRECISION

Repeatability:	0.12 X
Reproducibility:	0.22 X

Where X is the mean value.

There being no criteria for measuring bias in these test-product combinations, no statement of bias can be made.

OXYGEN IN GASOLINE AND FUELS BY REDUCTIVE PYROLYSIS:
D 5622

EXPLANATION

The presence of oxygen-containing compounds in gasoline can promote more complete combustion which reduces carbon monoxide emissions. The Clean Air Act of 1992 requires that gasoline sold within certain, specified geographical areas contains a minimum amount of oxygen (currently 2.7 mass %) during certain periods of the year. These test methods cover the determination of total oxygen in gasoline and methanol fuels. These test methods complement Test Method D 4815, which covers the determination of several specific oxygen-containing compounds in gasoline.

Several types of instruments can be used in these test methods. All pyrolyze the fuel in a carbon-rich environment. Instruments, however, differ in the way the oxygen-containing species are detected and quantitated.

TEST SUMMARY

A fuel sample is injected by syringe into a 950 to 1300°C high-temperature tube furnace that contains metallized carbon. Oxygen containing compounds are pyrolyzed, and the oxygen is quantitatively converted into carbon monoxide. A carrier gas such as N, He, or a He-H mixture, sweeps the pyrolysis gases into any of four downstream systems of reactors, scrubbers, separators, and detectors for the determination of the carbon monoxide content, and hence, of the oxygen in the original fuel sample.

TEST PRECISION

Oxygen, Mass %	Repeatability	Reproducibility
1.0 to 5.0	0.06 %	0.26 %
40 to 50	0.81 %	0.81 %

Based on the analysis of NIST SRM, this test method has no bias.

OXYGENATES IN GASOLINE BY GAS CHROMATOGRAPHY:
D 5599
(Equivalent Test Method: IP 408)

EXPLANATION

Alcohols, ethers, and other oxygenates are added to gasoline to increase the octane number and to reduce tailpipe emission of carbon monoxide. They must be added in the proper concentration and ratios to meet regulatory limitations and to avoid phase separation and problems with engine performance or efficiency. This test method provides sufficient oxygen-to-hydrocarbon selectivity and sensitivity to allow determination of oxygenates in gasoline samples without interference from the bulk hydrocarbon matrix. This test method is applicable to gasolines having a FBP not greater than 220°C and oxygenates having a boiling point limit of 130°C. It is applicable when oxygenates are present in the 0.1 to 20 mass % range.

This test method can determine the mass concentration of each oxygenate compound present in a gasoline. For calibration purposes, this requires knowledge of the identity of each oxygenate being determined. However, the oxygen-selective detector used exhibits a response to total amount of oxygen present whether individual compounds are identified or not.

Test Methods D 4815 and D 5845 for the determination of MTBE in gasoline have been described earlier. Test Method D 6293 for the determination of O-PONA hydrocarbons in fuels is described next.

TEST SUMMARY

An internal standard of a noninterfering oxygenate (for example, 1,2-dimethoxyethane) is added in a quantitative proportion to the gasoline sample. An aliquot of this mixture is injected into a gas chromatograph equipped with a capillary column operated to ensure separation of the oxygenates. Oxygenates are detected with the oxygen-selective flame ionization detector. Calibration mixtures are used to determine the retention times and relative mass response factors of the oxygenates of interest. The peak area of each oxygenate in the gasoline is measured relative to the peak area of the internal standard. The concentration of each oxygenate is calculated by using a quadratic least-squares fit of the calibration data of each oxygenate.

TEST PRECISION

See the precision in the following table.

The bias of this test method is being developed.

Repeatability for Oxygenates in Gasoline.

Component	Repeatability
Methanol (MeOH)	0.07 ($X^{0.49}$)[a]
Ethanol (EtOH)	0.03 ($X^{0.92}$)
Iso-propanol (iPA)	0.04 ($X^{0.54}$)
tert-Butanol (tBA)	0.05 ($X^{0.65}$)
n-Propanol (nPA)	0.04 ($X^{0.35}$)
MTBE	0.05 ($X^{0.58}$)
sec-Butanol (sBA)	0.03 ($X^{0.54}$)
DIPE	0.05 ($X^{0.65}$)
Iso-butanol (iBA)	0.03 ($X^{0.79}$)
ETBE	0.04 ($X^{0.86}$)
tert-Pentanol (tAA)	0.05 ($X^{0.41}$)
n-Butanol (nBA)	0.06 ($X^{0.46}$)
TAME	0.04 ($X^{0.58}$)
Total Oxygen	0.03 ($X^{0.93}$)

Reproducibility in Oxygenates in Gasolines.

Component	Reproducibility
Methanol (MeOH)	0.25 ($X^{0.86}$)
Ethanol (EtOH)	0.27 ($X^{0.80}$)
Iso-propanol (iPA)	0.21 ($X^{0.71}$)
tert-Butanol (tBA)	0.20 ($X^{0.80}$)
n-Propanol (nPA)	0.17 ($X^{0.88}$)
MTBE	0.10 ($X^{0.95}$)
sec-Butanol (sBA)	0.17 ($X^{0.73}$)
DIPE	0.16 ($X^{0.71}$)
Iso-butanol (iBA)	0.19 ($X^{0.83}$)
ETBE	0.25 ($X^{0.79}$)
tert-Pentanol (tAA)	0.18 ($X^{0.55}$)
n-Butanol (nBA)	0.22 ($X^{0.30}$)
TAME	0.24 ($X^{0.69}$)
Total Oxygen	0.13 ($X^{0.83}$)

[a] X is the mean mass % of the component.

OXYGENATES O-PONA HYDROCARBONS IN FUELS BY GC:

D 6293

EXPLANATION

A knowledge of spark-ignition engine fuel composition is useful for regulatory compliance, process control, and quality assurance. This test method provides for the quantitative determination of oxygenates, paraffins, olefins, napthenes, and aromatics in low-olefin spark-ignition engine fuels by multidimensional gas chromatography. The hydrocarbons can be reported by type through C_{10}. The lower limit of detection for a single hydrocarbon component or carbon number type is 0.05 mass %. This test method is applicable for total olefins in the range from 0.05 to 13 mass %. Although specifically written for spark-ignition engine fuels containing oxygenates, this test method can also be used for other hydrocarbon streams having similar boiling ranges, such as napthas and reformates. The test method is not applicable to M85 and E85 fuels, which contain 85 % methanol and ethanol, respectively.

TEST SUMMARY

A fuel sample is injected into a computer-controlled gas chromatographic system consisting of switching valves, columns, and an olefin hydrogenation catalyst, all operating at various temperatures. The eluted hydrocarbons are detected by a flame ionization detector. The mass concentration of the detected compounds is calculated from the response factors of the detectors and normalization to 100 %. Some sulfur compounds interfere by adsorbing on the columns. Commercial dyes and detergent additives added to the fuels, and dissolved water have been found not to interfere.

Repeatability and Reproducibility for Selected Oxygenate and Hydrocarbon Type Components and Groups of Components.[1]

			Range of Concentration	
Category	Repeatability	Reproducibility	Low	High
Aromatics	$0.0247 \cdot (4.3353 + X)$	$1.249 \cdot (4.3353 + X)$	12	44
Olefins	$0.046 \cdot X^{0.7444}$	$0.255 \cdot X^{0.7444}$	0.2	13
Paraffins	0.97	3.90	34	60
Naphthenes	$0.028 \cdot X$	$0.1659 \cdot X$	2	15

MTBE	$0.0155\cdot(1.858+X)$	$0.0641\cdot(1.858+X)$	0	12
Benzene	0.02	0.14	0.3	1
Toluene	$0.019\cdot X$	$0.0545\cdot X$	2	11
C_8 Aromatics	$0.0255\cdot(1.5172+X)$	$0.0708\cdot(1.5172+X)$	3.5	15

NOTE—1—X is the average of two results in mass % (or liquid volume %).

[1] This table is Table 1 from Test Method D 6293.

PARTICULATE CONTAMINATION IN AVIATION FUELS: D 5452

EXPLANATION

This test method provides a gravimetric measurement of the particulate matter present in a sample of aviation turbine fuel. The objective is to minimize these contaminants to avoid filter plugging and other operational problems. Although tolerable levels of particulates have not yet been established for all points in fuel distribution systems, the total contaminant measurement is normally of interest.

TEST SUMMARY

A known volume of fuel is filtered through a preweighed test membrane filter and the increase in membrane filter mass is weight determined after washing and drying. The change in weight of a control membrane located immediately below the test membrane filter is also determined. The particulate contaminant is determined from the increase in mass of the test membrane relative to the control membrane filter.

TEST PRECISION

Repeatability: $0.415X^{0.5}$
Reproducibility: $0.726X^{0.5}$

Where X is the mean of the two results.

This precision was determined in the range from 0 to 0.6 mg/L of particulate content.

This test method has no bias.

PARTICULATE CONTAMINATION IN AVIATION FUEL: D 2276

(Equivalent Test Method: IP 216)

EXPLANATION

See explanation in Test Method D 5452. This test method uses a field monitor. Two methods are described. One measures gravimetrically. The other uses a color rating technique for rapid qualitative assessment of changes in contaminant level without the time delay required for gravimetric determinations in the laboratory.

TEST SUMMARY

See Test Summary in Test Method D 5452. In the field monitoring test, a sample of fuel is taken from a flowing line or pipe and passed under line pressure through a field monitor containing a 0.8 μm test filter membrane. The color on the filter membrane is compared with the ASTM color standards and assigned a rating letter and number.

TEST PRECISION

Repeatability: $0.175X+0.070$
Reproducibility: $0.444X+0.178$

Where X is the average value of two results.

This precision is based on the range from 0.0 to 2.0 mg/L.

This test method has no bias.

PARTICULATE CONTAMINATION IN MIDDLE DISTILLATE FUELS:

D 6217

(Equivalent Test Methods: IP 415 and ISO 15167)

EXPLANATION

The mass of particulates present in a fuel is a significant factor, along with size and the nature of the individual particles, in the rapidity with which the fuel filters and other small orifices in fuel systems can become clogged. This test method provides such results. This test method can be used in specifications and purchase documents as a means of controlling particulate contamination levels in the purchased fuels. Several military fuel specifications specify maximum particulate levels in fuels. This test method is suitable for all No. 1 and 2 grades in Specifications D 396, D 975, D 2880, and D 3699, and for grades DMA and DMB in Specification D 2069. This test method is not suitable for fuels whose flash point as determined by Test Methods D 56, D 93, or D 3828 is less than 38°C.

TEST SUMMARY

A measured volume of about 1 L of the fuel sample is vacuum filtered through one or more sets of 0.8 μm membranes. Each membrane set consists of a tared nylon test membrane and a tared nylon control membrane. Depending upon the level of particulate contaminants in the sample, one or more than two membrane sets may be required. After the filtration has been completed, the membranes are washed with solvent, dried, and weighed.

TEST PRECISION

Repeatability:	$0.68X^{0.5}$
Reproducibility:	$1.13X^{0.5}$

Where X is the test result, measured to the nearest 0.1 g/m^3.

This precision is applicable to particulate contaminant levels between 0 to 25 g/m^3 provided that 1-L samples are used and the 1 L is filtered completely. Higher levels of particulates can be measured but are subject to uncertain precision. This test method has no bias.

PEROXIDES IN BUTADIENE:

D 5799

EXPLANATION

Due to inherent danger of peroxides in butadiene, specification limits are usually set for their presence. Butadiene polyperoxide is a very dangerous product of the reaction between butadiene and oxygen that can occur. The peroxide has been reported to be the cause of some violent explosions in vessels that are used to store butadiene. This test method will provide values that can be used to determine the peroxide content of a sample of commercial butadiene. This test method covers the concentration range of 1 to 10 ppm by mass as available oxygen.

TEST SUMMARY

A known mass of the butadiene sample is placed in a flask and evaporated. The residue is then refluxed with acetic acid and sodium iodide reagents. The peroxides react to liberate iodine which is titrated with standard sodium thiosulfate solution using visual end point detection. Interfering traces of iron are complexed with sodium fluoride.

TEST PRECISION

Repeatability:	1.4 ppmw
Reproducibility:	3.4 ppmw

As no reliable source of butadiene polyperoxide is available, the actual bias of this test method is unknown; but published data reports that this test method determines 90 % of the polyperoxide.

POLYCHLORINATED BIPHENYLS (PCBs) IN WASTES BY GAS CHROMATOGRAPHY:
D 6160

EXPLANATION

This test method provides sufficient PCB data for many regulatory requirements. While the most common regulatory level is 50 ppm, lower limits are used in some locations. This test method uses a two-tiered analytical approach to PCB screening and quantitation of liquid and solid wastes, such as oils, sludges, aqueous solutions, and other waste matrices. Tier I is designed to rapidly screen the samples for the presence of PCBs. Tier II is used to determine their concentration, typically in the range 2 to 50 ppm. Greater concentrations can be determined through sample dilutions. Quantification limits will vary depending on the types of waste streams being analyzed.

TEST SUMMARY

The sample is extracted with solvent and the extract is treated to remove interfering substances, if needed. The extract is injected into a gas chromatograph. The components are separated as they pass through the capillary column and PCB compounds, if present, are detected by an electron capture detector (ECD). For quantification, an external standard—Aroclor—is used. The ECD has selective sensitivity to many other compounds; hence, the chromatograms obtained need to be carefully checked. Solvents, reagents, glassware, etc., may produce artifacts in the analysis. Phthalates interfere, hence, all contact of samples and extracts with plastic should be avoided. Other detectors, such as atomic emission, or mass spectrometry may be used, if sufficient performance is demonstrated.

TEST PRECISION

Repeatability:	$0.16X^{1.1}$
Reproducibility:	$0.73X^{1.1}$

Where X is average PCB concentration in mg/kg.

This precision is based on data collected by GC/ECD.

A reliable quantitation of bias for this method was not possible. However, the method tends to produce low results. This tendency is mitigated to some extent through the use of a surrogate.

PEROXIDE NUMBER OF AVIATION TURBINE FUELS:
D 3703

EXPLANATION

The magnitude of the peroxide number is an indication of the quantity of oxidizing constituents present in the sample. Deterioration of turbine fuel results in the formation of the peroxides and other oxygen-carrying compounds. The peroxide number measures those compounds that will oxidize potassium iodide. The determination of peroxide number of aviation turbine fuel is important because of the adverse effects of peroxides upon certain elastomers in the fuel system.

TEST SUMMARY

The sample is dissolved in 1,1,2-trichloro-1,2,2-trifluoroethane, and is contacted within an aqueous potassium iodide solution. The peroxides present are reduced by the potassium iodide. An equivalent amount of iodine is released which is titrated with standard sodium thiosulfate solution using a starch indicator.

TEST PRECISION

Repeatability:	$0.15X$
Reproducibility:	Not determined

Bias has not been determined.

PEROXIDE NUMBER OF PETROLEUM WAX: D 1832

EXPLANATION

Deterioration of petroleum wax results in the formation of peroxides and other oxygen-containing compounds. The peroxide number measures those compounds that will oxidize potassium iodide. Thus the magnitude of the peroxide number is an indication of the quantity of oxidizing constituents present.

TEST SUMMARY

A sample is dissolved in xylenes and is acidified with acetic acid. A solution of potassium iodide is added, and after a reaction period, the solution is titrated with sodium thiosulfate using a starch indicator.

TEST PRECISION

This test method has a repeatability of 1.5 and a reproducibility of 3.3 in the peroxide number range of 0 to 15.

This test method has no bias.

PETROLEUM WAX ANALYSIS BY GC: D 5442

EXPLANATION

The determination of carbon number distribution of petroleum waxes and the normal and nonnormal hydrocarbons in each can be used for controlling the production processes as well as a guide to performance in many end users, particularly in rubber formulations. This test method determines n-C_{17} through n-C_{44} by gas chromatography with internal standardization. Material with a carbon number above n-C_{44} is determined by difference from 100 mass % and is reported as C_{45}+. This test method is applicable to petroleum derived waxes and wax blends. It is, however, not applicable to oxygenated waxes such as synthetic polyethylene glycols, or natural products such as beeswax or carnauba. This test method is not directly applicable to waxes with an oil content greater than 10 % as determined by Test Method D 721.

TEST SUMMARY

A sample of petroleum wax and an internal standard are completely dissolved in an appropriate solvent and injected into a gas chromatographic column that separates the hydrocarbons by increasing carbon number. The column temperature is linearly increased at a reproducible rate until the sample is completely eluted from the column. The eluted components are detected by a flame ionization detector. The individual carbon numbers are identified by comparing the retention times obtained from a qualitative standard with the retention times of the wax sample. The percentage of carbon number through 44 is calculated via internal standard calculations after applying response factors.

TEST PRECISION[1]

See the precision estimates in the following table.

Repeatability and Reproducibility.

Carbon Number	Range, mass %	Repeatability[a]	Reproducibility[a]
C_{21}	0.11–0.25	0.014	0.039
C_{23}	0.04–2.90	$0.0463\ X^{0.30}$	$0.1663\ X^{0.30}$
C_{26}	0.01–8.94	$0.0785\ X^{0.56}$	$0.4557\ X^{0.56}$
C_{29}	0.04–8.15	$0.0872\ X^{0.31}$	$0.3984\ X^{0.62}$
C_{32}	0.44–5.05	$0.1038\ X^{0.50}$	$0.6472\ X^{0.50}$
C_{35}	2.52–5.62	$0.1737\ X$	$0.4540\ X$
C_{38}	0.44–3.61	$0.1131\ (X+0.1069)$	$0.5476\ (X+0.1069)$
C_{41}	0.06–2.96	$0.1600\ X$	$0.5460\ X$
C_{44}	0.02–2.26	$0.4990\ X^{0.60}$	$0.9220\ X^{0.60}$
Total *n*-paraffins	18.73–79.52	2.64	26.03

[a] Where$X$ is the mass % of the component.

[1] This table is Table 2 from Test Method D 5442.

The bias of this method has not been determined.

PHOSPHORUS DETERMINATION IN PETROLEUM PRODUCTS:

GENERAL

Depending upon the product, phosphorus can serve as a beneficial adjunct or as a deleterious agent. Phosphorus in gasoline will damage catalytic converters used in automotive emission control systems, and its level therefore is kept low. In additives and lubricating oils, phosphorus is added as an organophosphorus compound to improve engine performance.

There are several test methods for the determination of phosphorus. In addition to the three test methods described here, reference should also be made to multielement analysis methods such as ICPAES (Test Methods D 4951 and D 5185) and XRF (Test Methods D 4927 and D 6443) described earlier in this guide.

PHOSPHORUS DETERMINATION IN GASOLINE:

D 3231

TEST SUMMARY

This test method is applicable for the determination of phosphorus in the range from 0.2 to 40 mg/L. Organic matter in the sample is decomposed by ignition in the presence of zinc oxide. The residue is dissolved in sulfuric acid and reacted with ammonium molybdate and hydrazine sulfate. The absorbance of molybdenum blue complex is measured at 820 nm in a 5-mm cell in a spectrophotometer, and is proportional to the phosphorus concentration in the sample.

TEST PRECISION

P Range, mg/L	Repeatability	Reproducibility
0.2 to 1.3	0.05	0.13
1.3 to 40	7 % of the Mean	13 % of the Mean

Bias of this test method has not been determined.

PHOSPHORUS DETERMINATION IN LUBRICATING OILS: D 1091

TEST SUMMARY

Organic material in the sample is destroyed and phosphorus in the sample is converted to phosphate ion by oxidation with sulfuric acid + nitric acid + hydrogen peroxide. When the phosphorus content is <2 %, the molybdovanado photometric method is used; when the phosphorus content is >2 %, the magnesium pyrophosphate gravimetric method is used.

TEST PRECISION

See Figs. 13–16.

The bias of this test method has not been determined.

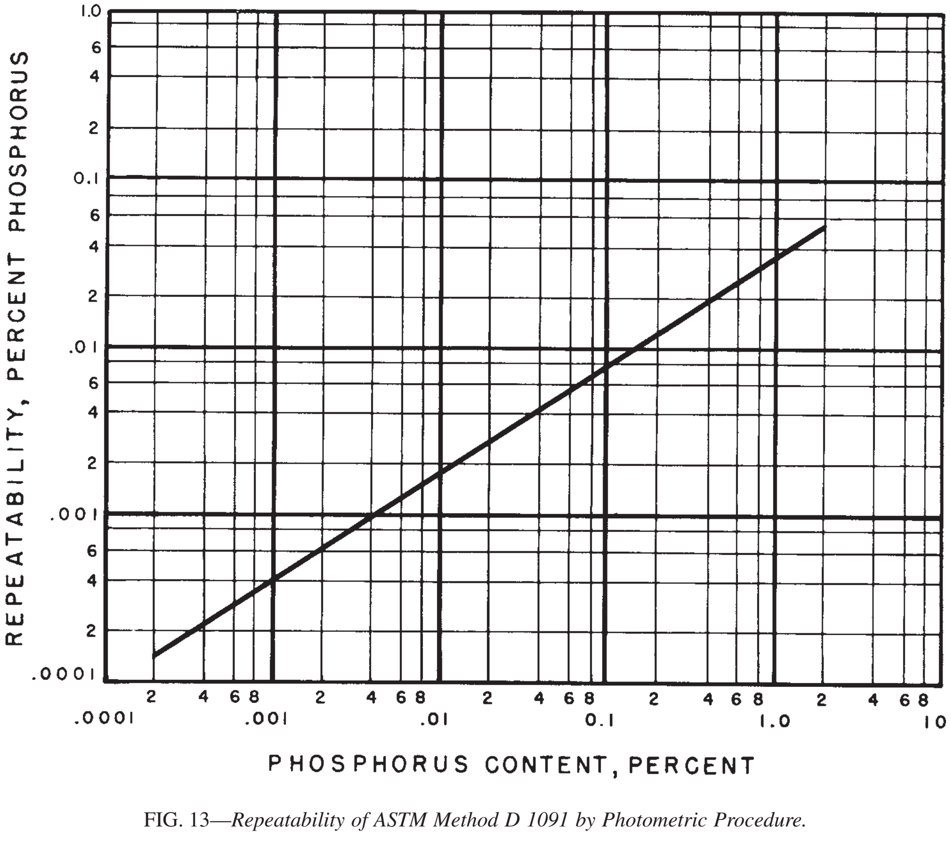

FIG. 13—*Repeatability of ASTM Method D 1091 by Photometric Procedure.*

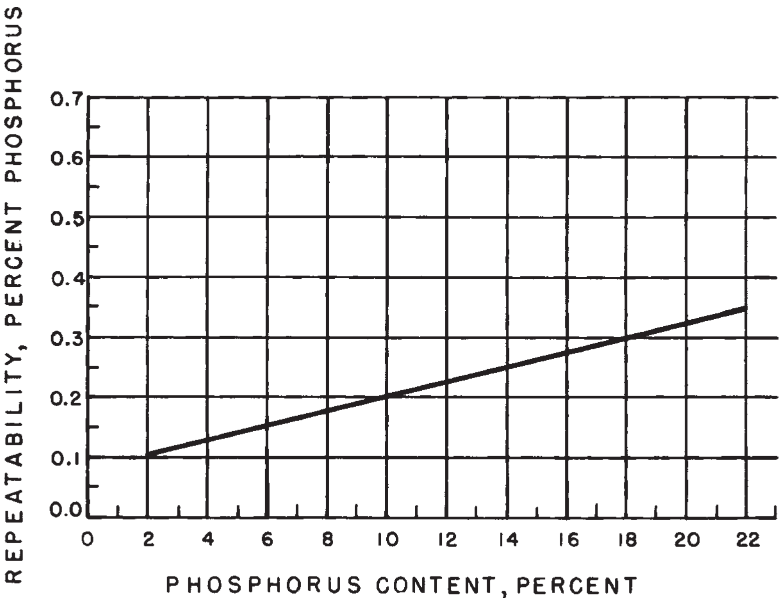

FIG. 14—*Repeatability of ASTM Method D 1091 by Gravimetric Procedure.*

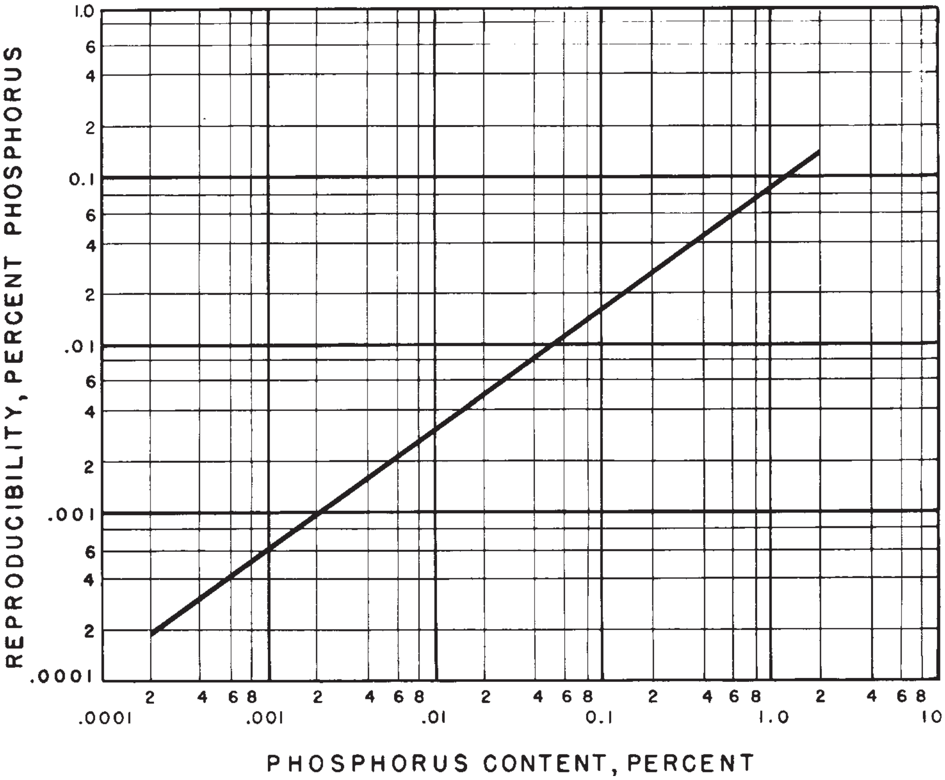

FIG. 15—*Reproducibility of ASTM Method D 1091 by Photometric Procedure.*

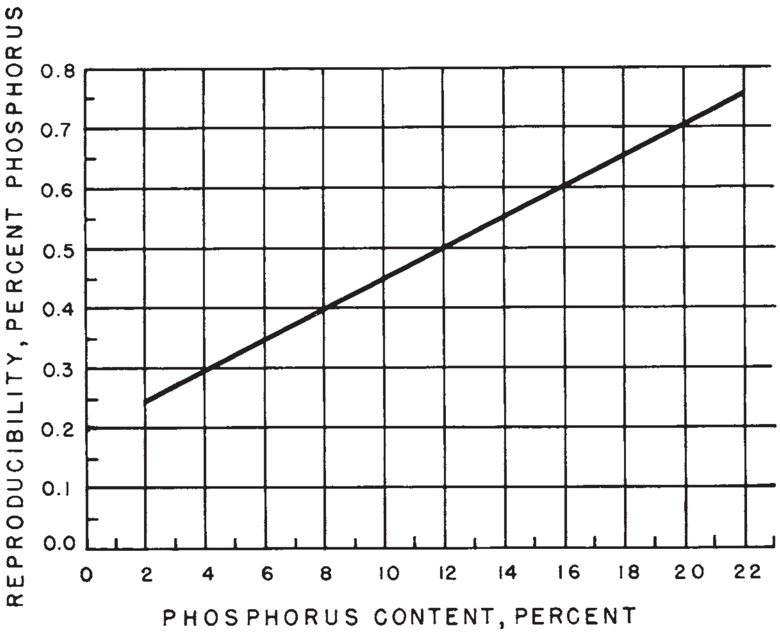

FIG. 16—*Reproducibility of ASTM Method D 1091 by Gravimetric Procedure.*

PHOSPHORUS DETERMINATION IN LUBRICATING OILS: **D 4047**

(Equivalent Test Methods: IP 149 and ISO 4265)

TEST SUMMARY

This test method is applicable in the range 0.005 to 10.0 mass % of phosphorus in unused lubricating oils and additive concentrates. It should be applicable to filtered used oils also; but no study on this has been done. The sample is ignited with an excess of zinc oxide whereby phosphorus is converted to phosphate. The residue is dissolved in hydrochloric acid, and any sulfide formed is oxidized with potassium bromate. Phosphorus is then precipitated as quinoline phosphomolybdate and determined volumetrically by addition of excess standard alkali and back titration with standard acid.

TEST PRECISION

Phosphorus, mass %	0.005–0.079	0.08–10
Repeatability:	$0.032(X+0.04)$	$0.0318X^{0.992}$
Reproducibility:	$0.074(X+0.04)$	$0.118X^{0.992}$

Where X is the average of two results.

The bias of this test method is not known.

PISTON DEPOSITS BY TEOST MHT: **D 7097**

EXPLANATION

This test method is designed to predict the deposit-forming tendencies of engine oils in the piston ring belt and upper piston crown area. Correlation has been shown between the TEOST MHT procedure and the TU3MH Peugot engine test in deposit formation. Such deposits formed in the ring belt area of a reciprocating engine piston can cause problems with engine operation and longevity. It is one of the required test methods in Specification D 4485 to define API Category Identified engine oils. This test method covers the procedure to determine the mass of deposit formed on a specially constructed test rod exposed to repetitive passage of 8.5 g of engine oil over the rod in a thin film under oxidative and catalytic conditions at 285°C. The range of applicability of the Moderately High Temperature Thermo-Oxidation Engine Test (TEOST MHT) test method as derived from an interlaboratory study is approximately 10 to 100 mg. However, experience indicates that deposit values from 1–150 mg or greater may be obtained. This test method uses a patented instrument, method and patented, numbered, and registered depositor rods traceable to the manufacturer, and made specially for the practice and precision of the test method.

TEST SUMMARY

Deposit-forming tendencies of an engine oil under oxidative conditions are determined by calculating an oil-catalyst mixture comprising a small sample (8.4 g) of the oil and a very small (0.1 g) amount of an organo-metallic catalyst. This sample mixture is then circulated for exactly 24 hours in the TEOST MHT instrument over a special wire-wound depositor rod heated by electrical current to a controlled temperature of 285°C at the hottest location on the rod. The depositor rod is weighed before and after the test, and any deposit formation on the rod as well as any deposits collected from rod washings are determined. During the test, precisely controlled and directed air is caused to bath the oil flowing down the depositor rod and, thereby, to provide opportunity for oxidation. Precision of this test is strongly influenced by care in manufacture of the wire-wound steel depositor rods and the treatment of the coating of the wound wire, the rate of air flow, and the amount and degree of mixing of the catalyst.

TEST PRECISION

Repeatability:	$0.15(x+16)$
Reproducibility:	$0.35(x+16)$

Where x is the mean of two test results.

No information on the bias of this method can be presented because no material having an accepted reference value is available.

POUR POINT OF CRUDE OILS: D 5853

EXPLANATION

The pour point of a crude oil is an index of the lowest temperature of handleability for certain applications. The maximum and minimum pour point temperatures provide a temperature window where a crude oil, depending on its thermal history, might appear in the liquid as well as the solid state. This test method can be used to supplement other measurements of cold flow behavior. It is especially useful for the screening of the effect of wax interaction modifiers on the flow behavior of the crude oils. This test method covers two procedures for the determination of the pour point of crude oils down to −36°C. One method provides a measure of the maximum (upper) pour point temperature. The second method measures the minimum (lower) pour point temperature.

TEST SUMMARY

After preliminary heating, the test specimen is cooled at a specified rate and examined at intervals of 3°C for flow characteristics. The lowest temperature at which movement of the test specimen is observed is recorded as the pour point.

TEST PRECISION

Procedure	Repeatability, °C	Reproducibility, °C
A	3.1	18.0
B	5.8	22.0

This procedure has no bias.

TABLE 10—*Test Methods For The Determination of Pour Point in Petroleum Products.*

Test Method	Technique	Scope	Limitations	Repeatability	Reproducibility	Bias
D 97	Manual. Lowest temperature at which movement of cooled oil is noted	Petroleum products	Measured in 3°C intervals	3°C	6°C	N/A
D 5853	Same as above	Crude oils	Measured in 3°C intervals	A: 3.1°C B: 5.8°C	A: 18°C B: 22°C	N/A
D 5949	Automatic method. Optical device used to detect movement of a Cooling Sample (Phase Technology)	−57 to +51°C	Not for crude oils	3.4°C	6.8°C	N/A
D 5950	Automatic method. Sample jar is tilted as it cools to detect surface movement with an optical device (ISL)	−57 to +51°C Measurement at 1, 2, or 3°C intervals possible	Not for crude oils	4.4°C	4.8°C	N/A
D 5985	Automatic method. Sample is rotated while cooling and crystal formation or viscosity increase is detected (Herzog)	−57 to +51°C	Not for crude oils	2.3°C	8.7°C	N/A
D 6749	Automatic method. Sample is cooled and air pressure is used to detect specimen surface with pressure sensor (Tanaka)	−57 to +51°C Results similar to those by Test Method D 97. Results in 1 or 3°C intervals possible	Not for crude oils or residual fuels	@ 1°C: 1.1; @ 3°C: 2.5	@ 1°C: 2.2; @ 3°C: 3.1	Variable
D 6892	Automatic method. Jar is tilted till surface movement is detected with optical camera (Herzog)	−57 to +51°C 1 to 3°C intervals possible. Results similar to Test Method D 97	Not for crude oils or residual fuels	@ 1°C: 1.8; @ 3°C: 3.2	@ 1°C: 3.2; @ 3°C: 3.6	Variable

POUR POINT:
D 97
(Equivalent Test Methods: IP 15, ISO 3016, DIN 51597, JIS K 2269, and AFNOR T60-105)

EXPLANATION

Pour point of a petroleum oil is an index of the lowest temperature of its utility for certain applications. The cloud point (Test Method D 2500) is usually higher than the pour point.

TEST SUMMARY

After preliminary heating, the sample is cooled at a specified rate and examined at intervals of 3°C for flow characteristics. The lowest temperature at which the movement of the oil is observed is recorded as the pour point.

TEST PRECISION

Repeatability:	3°C
Reproducibility:	6°C

NOTE

Three new pour point test methods are now available for Herzog, IT Phase Technology, and ATPEM automatic machines. The three methods show somewhat different precisions, but show no real bias against Test Method D 97, which will be considered the referee method in case of a dispute.

POUR POINT OF PETROLEUM PRODUCTS BY USING AUTOMATIC AIR PRESSURE METHOD:
D 6749

EXPLANATION

The pour point of a petroleum product is an index of the lowest temperature of its utility for certain applications. Flow characteristics, like pour point, can be critical for the correct operation of lubricating systems, fuel systems, and pipeline operations. Test results from this method can be determined at either 1 or 3°C intervals. This test method yields a pour point in a method similar to Test Method D 97 when the 3°C intervals are reported. This test method has better precision relative to Test Method D 97 as measured in the 1998 interlaboratory test program. However, do not substitute this method when specifications require Test Method D 97. This test method is designed to cover the temperature range from −57 to +51°C. This test method is not intended to use for crude oils nor its applicability verified for residual fuels.

TEST SUMMARY

After inserting the test jar containing the specimen into the automatic pour point apparatus and initiating the test program, the specimen is automatically heated to the designated temperature and then cooled at a controlled rate. At temperature intervals of 1 or 3°C, depending on the selection made by the user prior to the test, a slightly positive air pressure is gently applied onto the surface of the specimen which is contained in an air-tight test jar equipped with a communicating tube. Since one end of the communicating tube is inserted into the specimen, while the other end is maintained at atmospheric pressure, a small amount of downward movement or deformation of the specimen surface, as a result of the application of air pressure, is observed by means of upward movement of the specimen in the communicating tube. This upward movement of the specimen is detected by a pressure sensor which is installed at the atmospheric end of the communicating tube. The lowest temperature at which deformation of the specimen is observed upon application of air pressure is recorded as the pour point.

TEST PRECISION

Testing Interval, °C	1	3
Repeatability	1.1	2.5
Reproducibility	2.2	3.1

Relative bias among certain samples was observed using 3°C intervals. However, it does not appear to be of a systematic nature. A relative bias of 1.1°C was observed when comparing testing at 1 versus 3°C intervals.

POUR POINT AUTO POUR POINT (PHASE TECHNOLOGY):
D 5949

EXPLANATION

This test method covers the determination of pour point of petroleum products by an automatic instrument that applies a controlled burst of nitrogen gas onto the specimen surface while the specimen is being cooled and detects movement of the surface of the test specimen with an optical device. This test method includes the range of temperatures from −57 to +51°C. Test results from this test method can also be determined at 1 and 3°C testing intervals; however, precision data of these testing intervals are not available. This test method is not intended for use with crude oil. The applicability of this test method has not been verified for residual fuel samples.

TEST SUMMARY

After inserting the test specimen into the automatic pour point apparatus, and initiation of the test program, the test specimen is heated and then cooled by a Peltier device at a rate of 1.5±0.1°C/min. At temperature intervals of 1, 2 or 3°C, depending on the selection made by the user, a moving force in the form of a pressurized pulse of nitrogen gas is imparted onto the surface of the specimen. Multiple optical detectors are used in conjunction with a light source to monitor movement of the surface of the specimen. The lowest temperature at which movement of the specimen surface is observed upon application of a pulse of nitrogen gas is recorded as the pour point, Test Method D 5949.

TEST PRECISION

	1°C Test Interval		3°C Test Interval	
	Repeatability	Reproducibility	Repeatability	Reproducibility
Model 70V	1.6°C	3.2°C	2.2°C	3.8°C
Models 30, 50, 70	2.9°C	6.2°C	4.1°C	6.3°C

POUR POINT AUTO POUR POINT (ISL):
D 5950

EXPLANATION

This test method covers the determination of pour point of petroleum products by an automatic instrument that tilts the test jar during cooling and detects movement of the surface of the test specimen with an optical device. This test method includes the range of temperatures from −57 to +51°C. Test results from this test method can be determined at 1 or 3°C intervals. This test method is not intended for use with crude oils. The applicability of this test method has not been verified for residual fuel samples.

TEST SUMMARY

After preliminary heating, the test specimen is inserted into the automatic pour point apparatus. After starting the program, the specimen is cooled according to the cooling profile listed in Table 1 of Test Method D 5950 and examined in either 1, 2, or 3°C intervals. The lowest temperature at which movement of the specimen is detected, by the automatic equipment, is displayed as the pour point.

TEST PRECISION

	1°C Testing Interval	3°C Testing Interval
Repeatability	2.7°C	3.9°C
Reproducibility	4.5°C	6.1°C

POUR POINT OF PETROLEUM PRODUCTS BY ROBOTIC TILT METHOD:

D 6892

EXPLANATION

See explanation in Test Method D 6749 earlier. Test results from this method can be determined at either 1 or 3°C intervals. This test method yields a pour point in a format similar to Test Method D 97, when the 3°C interval results are reported. This test method has comparable repeatability and better reproducibility relative to Test Method D 97. In this test method the automatic instrument tilts the test jar to detect surface movement of the test specimen with an optical device, after being removed from a regulated, stepped-bath cooling jacket. This test method is designed to cover the temperature range of −57 to +51°C. This test method is not intended for use with crude oils, nor its applicability has not been verified for residual fuels.

TEST SUMMARY

After insertion of the specimen into the automatic pour point apparatus and initiation of the testing program, the specimen is heated and then cooled according to a prescribed profile. The specimen surface is examined periodically for movement using an optical camera system mounted on top of the specimen test jar, while tilting the specimen test jar. The test jar is removed from the jacketed cooling chamber prior to each examination. The lowest temperature, when movement of the surface of the specimen is detected, is recorded as the pour point.

TEST PRECISION

Testing Intervals, °C:	1	3
Repeatability:	1.8	2.3
Reproducibility:	3.2	3.6

When testing at 3°C intervals, relative bias among certain samples was observed versus results from Test Method D 97. The relative bias was not a fixed value but appeared to be a linear function of the pour point value. When measuring at 1°C intervals, a bias of 1.1°C on the average was observed against Test Method D 97.

POUR POINT AUTO POUR POINT (HERZOG):

D 5985

EXPLANATION

This test method covers the determination of pour point of petroleum products by an automatic instrument and continuously rotates the test specimen against a suspended detection device during cooling of the test specimen. This test method includes the range of temperatures from −57 to +51°C. This test method determines the no-flow point of petroleum products by detection of the crystal structure or viscosity increase, or both, in the sample that is sufficient to impede flow of the specimen. This test method is not intended for use with crude oils. The applicability of this test method to residual fuels oils has not been verified.

TEST SUMMARY

After inserting the test specimen into the automatic pour point apparatus, and initiation of the program, the test specimen is heated and then cooled by maintaining a constant temperature differential between the cooling block and the sample. The test specimen is continuously tested for flow characteristics by rotating the test specimen cup at approximately 0.1 r/min against a stationary, counterbalanced, sphere-shaped pendulum. The temperature of the test specimen at which a crystal structure or a viscosity increase, or both, within the test specimen causes the displacement of the pendulum and is recorded with a resolution of 0.1°C. The test specimen is then heated to the original starting temperature.

TEST PRECISION

Repeatability:	2.3°C
Reproducibility:	8.7°C

PRECIPITATION NUMBER OF LUBRICATING OILS: D 91

EXPLANATION

Fully refined petroleum oils normally contain no naptha insoluble material. Semirefined or black oils frequently contain some naptha insoluble material, sometimes referred to as asphaltenes. This test measures the amount of naptha insoluble material in the steam cylinder stocks, black oils, and lubricating oils. This quantity is reported as the precipitation number.

TEST SUMMARY

Ten mL of lubricating oil is mixed with 90 mL of ASTM precipitation naptha, and centrifuged for 10 mm at a rcf of between 600 and 700. Volume on the bottom of the centrifuge cone is read until repeat centrifugation gives a reading within 0.1 mL. This reading is reported as the precipitation number.

TEST PRECISION

In the precipitation number range of 0.00 to 1.20, the test repeatability is 10 % of the mean, and the test reproducibility is 30 % of the mean.

This procedure is empirical, and no statement of bias can be made.

PUMPABILITY OF INDUSTRIAL FUEL OILS: D 3245

EXPLANATION

This test method is designed to give an indication of the minimum storage and minimum handling temperatures, which may be used for a given fuel oil. This test method is cited in Specification D 396.

TEST SUMMARY

A sample of oil, preheated if necessary to make it fluid, is poured into the cup of the portable viscometer. This is immersed in a bath at a predetermined temperature for 15 min. Then the viscometer is started at the shear rate of 9.7 s^{-1}. After a further 5 min, the bath is cooled at 0.5°C/min. The temperature at which apparent viscosities of 0.6 Pa.s and 2.5 Pa.s are obtained is determined.

TEST PRECISION

Temperature	Repeatability	Reproducibility
0.6 Pa.s	0.5°C	2.9°C
2.5 Pa.s	0.5°C	4.0°C

No statement of bias can be made for this test method.

QUENCHING TIME OF HEAT TREATING FLUIDS: D 3520

EXPLANATION

This method covers a procedure for making a comparative evaluation of the quenching speed characteristics of petroleum fluids used in quenching of metals by means of the magnetic quenchometer. A comparison method for evaluation of the quenching speed characteristics of petroleum fluids used in quenching of metals is Test Method D 6200. A comparison method for testing the hardenability of steel is Test Method A 255. Although this test method has been found useful for some water-based fluids, the statistical significance of the test has been established only by round robin testing of petroleum based fluids.

This test method provides a measure of changes in oil chemistry due to contamination, base oil degradation, and additive drag-out during use. This test measures changes in the quenching speed characteristics of petroleum fluids at the high temperature >354°C. The test method does not reliably predict metallurgical performance. Results from this test method provide a measure of quenching speed (cooling rate) from approximately 885°C–354°C, and are not directly proportional to hardness obtainable on metals quenched therein as many other factors are involved in the quenching process in actual plant operation with production parts.

TEST SUMMARY

This test method determines the time for cooling a chromized nickel ball from approximately 885°C to approximately 354°C when quenched in 200 mL of test fluid in a metal beaker at 21–27°C. The quenching time is recorded by a digital timer which is energized by a photoelectric cell from light produced by the ball at 885°C, and which is stopped when the ball becomes magnetic (Curie point, approximately 354°C), and is attracted by a magnet to the side of the beaker, tripping a relay to stop the timer. Test Method D 6200 describes the equipment and the procedure for characterizing the time-temperature performance of a quenching oil through the entire cooling process.

TEST PRECISION

Repeatability:	5 %
Reproducibility:	9 %

The values are means of quench time in seconds.

This method has no bias because the value of the quenching speed can be defined only in terms of this test method.

QUINOLINE INSOLUBLE CONTENT OF TAR AND PITCH:
D 2318

EXPLANATION

This test method is useful in evaluating and characterizing tar and pitch, and as one element in establishing the uniformity of shipments and sources of supply. Since this method is empirical, strict adherence to all details of the procedure is necessary.

TEST SUMMARY

The sample is digested in hot quinoline and filtered. The insoluble material is washed, dried, and weighed.

TEST PRECISION

Quinoline Insoluble:	4 % or less	More than 4 %
Repeatability:	0.2	0.3
Reproducibility:	0.3	0.6

This test method has no bias because the value of quinoline insoluble is defined in terms of this test method.

QUINOLINE INSOLUBLE IN TAR AND PITCH BY PRESSURE FILTRATION:
D 4746

EXPLANATION

See Explanation under Test Method D 2318 above. Both of these test methods give comparable results.

TEST SUMMARY

The sample is digested in hot quinoline and filtered through a heated pressure filter. The insoluble material is washed with hot, fresh quinoline and with cold acetone, dried and weighed.

TEST PRECISION

Repeatability:	0.2 %
Reproducibility:	0.3 %

RAMSBOTTOM CARBON RESIDUE: **D 524** (Equivalent Test Methods: IP 14, ISO 4262, and AFNOR T60-117)

EXPLANATION

The carbon residue value of burner fuel serves as a rough approximation of the tendency of the fuel to form deposits in vaporizing pot- and sleeve-type burners. Similarly, provided alkyl nitrates are absent, the carbon residue of the diesel fuel correlates approximately with combustion chamber deposits. The carbon residue value of motor oils, while once considered useful, is now considered to be of doubtful value because of the presence of additives in many oils. The carbon residue of gas oil is useful as a guide in the manufacture of gas from gas oil, while the carbon residue value of crude oil residuums, cylinder and bright stocks, are useful in the manufacture of lubricants. This test method is generally applicable to relatively non-volatile petroleum products which partially decompose on distillation at atmospheric pressure.

The values obtained by this method are not numerically the same as those obtained by Test Methods D 189 or D 4530.

TEST SUMMARY

The sample is weighed into a special glass bulb having a capillary opening, and is placed in a metal furnace maintained at about 550°C. In this process all volatile matter is evaporated out of the bulb, and the heavier residue remaining in the bulb undergoes cracking and coking reactions. After a specified heating period, the bulb is removed from the bath, cooled in a desiccator, and again weighed. The residue remaining is calculated as a percentage of the original sample and reported as Ramsbottom carbon residue.

Correlation with D189—See the discussion under D 189 Conradson Carbon (page to come).

TEST PRECISION

See Fig. 17 for the precision estimates.

This test method is empirical and no statement of bias can be made.

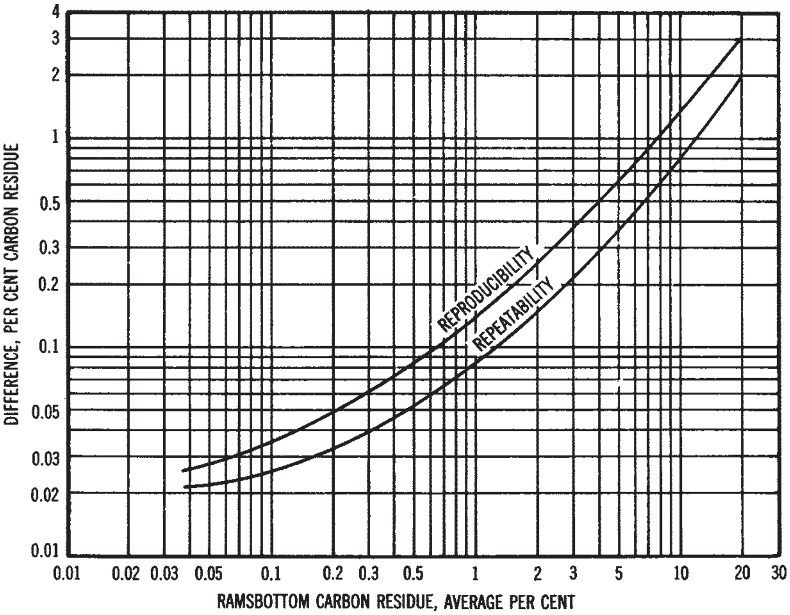

FIG. 17—*Precision Data.*

RED DYE CONCENTRATION AND ESTIMATION OF ASTM COLOR OF DIESEL FUEL AND HEATING OIL:
D 6756

EXPLANATION

In the United States, high sulfur content (as defined by the U.S. Environmental Protection Agency) middle distillate products and diesel fuel used for off-road purposes, other than aviation turbine fuels, are required by government agencies to contain red dye. The dye concentration required to be present in high-sulfur and off-road diesel products is regulated by USEPA, and the U.S. Internal Revenue Service, respectively.

Some fuels that are readily exchanged in the market have a color specification. The color of the base fuel is masked by the presence of the red dye. This test method provides a means of estimating the base color of Number 1 and Number 2 diesel fuel and heating oil in presence of red dye. The test method provides a means to indicate conformance to contractual and legal requirements. The test method is appropriate for use with diesel fuel and heating oil of Grades 1 and 2 described in Specifications D 396, D 975, D 2880, and D 3699. Red dye concentrations are determined at levels equivalent to 0.1–20 mg/L of Solvent Red 26 in samples with ASTM color ranging from 0.5–5. The ASTM color of the base fuel red-dyed samples with concentration levels equivalent to 0.1–20 mg/L of Solvent Red 26 is estimated for the ASTM color range from 0.5–5. The ASTM color of the undyed samples is estimated over the color range of 0.5–5.

TEST SUMMARY

A sample is introduced into the liquid specimen cell which is then placed into the light path of the apparatus. A beam of visible light is imaged through the sample onto a detector, and the detector response is determined. Wavelengths of the spectrum, which correlate highly with the red dye concentration and the ASTM color, are selected for analysis using selective bandpass filters. A multivariate mathematical model converts the absorption values from the filters to the red dye concentration and the estimated ASTM color.

INTERFERENCES—The presence of colorants other than the hydrocarbons typical of Number 1 and 2 diesel fuel and heating oils, or the presence of the red dye other than the specified types, can interfere with the accurate determination of the red dye concentration reported as Solvent Red 26 equivalent or the base fuel ASTM color.

TEST PRECISION

Parameter:	Solvent Red 26 Equivalent Dye concentration between 0.1 and 20.0 mg/L	ASTM color range of 0 to 5
Repeatability:	0.22 mg/L	0.085 units
Reproducibility:	0.86 mg/L	0.20 units

Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, bias has not been determined.

RED DYE CONCENTRATION AND ESTIMATION OF SAYBOLT COLOR OF AVIATION TURBINE FUELS AND KEROSENE:
D 7058

EXPLANATION

In the United States, high-sulfur content distillate products and diesel fuel used for off-road purposes, other than aviation turbine fuel are required to contain red dye. A similar dye requirement exists for tax-free distillates. Contamination of aviation turbine fuel by small quantities of red dye has occurred. Such contamination presents major problems because airframe and engine manufacturers have severely limited operation on aviation turbine fuel containing red dye.

An alternate methodology for the determination of the presence of the red dye in aviation turbine fuel is the observation of the color of the fuel when placed in a white bucket. The presence of the dye can be masked in the aviation turbine fuels having dark Saybolt color. This test method provides an objective means of quickly measuring red dye concentration, but to avoid confusion with trace levels of other materials which will be indicated by the instrument, the method requires that the instrument readings below 0.026 mg/L be reported as No Dye Present. The color of the base fuel is masked by the presence of the red dye. This test method provides a means of estimating the base color of aviation turbine fuel and kerosene in the presence of red dye. This test method covers the determination of the red dye concentration of aviation turbine fuel and kerosene and the estimation of the Saybolt color of undyed and red dyed (<0.750 mg/L of Solvent Red 26 equivalent) aviation turbine fuel and kerosene. The method is appropriate for use with aviation turbine fuel and kerosene described in Specifications D 1655 and D 3699. Red dye concentrations are determined at levels equivalent to 0.026 to 0.750 mg/L of Solvent Red 26 in samples with Saybolt colors ranging from +30 to −16. The Saybolt color of base fuel for samples dyed red with concentration levels equivalent to 0.026 to 0.750 mg/L of Solvent Red 26 is estimated in the Saybolt color range +30 to −16. The Saybolt color for undyed samples is estimated in the Saybolt color range from +30 to −16.

TEST SUMMARY

See the Test Method Summary under Test Method D 6756 on previous page; the only difference is that in D 6756 ASTM color equivalent is measured; in Test Method D 7058, Saybolt color equivalent is measured.

INTERFERENCES—The presence of colorants resulting from the refining process or crude oil or the presence of red dye other than the quantified types (alkyl derivatives of azobenzene-4-azo-2-naphthol) can interfere with the accurate determination of the red dye concentration reported as Solvent Red 26 equivalent, or the accurate estimation of the base fuel color. If there is controversy over whether the indicated dye concentration is from the alkyl derivatives of azobenzene-4-azo-2-naphthol, the procedure described in the Annex of this test method shall be used to confirm the presence of a red dye.

TEST PRECISION

Test Parameter:	Solvent Red 26 Equivalent Dye concentration in the Range 0 to 0.750 mg/L	Saybolt color range of −16 to +30
Repeatability:	0.006 mg/L	1.1 unit
Reproducibility:	0.026 mg/L	4.6 unit

Since there is no accepted reference material suitable for determining the bias of the procedure in this test method, bias has not been determined.

REFRACTIVE INDEX OF HYDROCARBON LIQUIDS: **D 1218**
(Equivalent Test Methods: ISO 5661 and DIN 51423 T2)

EXPLANATION

Refractive index and refractive dispersion are fundamental physical properties which can be used in conjunction with other properties to characterize pure hydrocarbons and their mixtures. This test method covers the measurement of refractive indices, accurate to six units in the fifth decimal place, of transparent and light-colored hydrocarbons that have refractive indices in the range from 20 to 30°C. The test is not applicable within the accuracy stated to liquids having colors darker than No. 4 ASTM color as determined by Test Method D 1500, to liquids having bubble points so near the test temperature that a reading cannot be obtained before substantial weathering takes place, to liquids having a refractive index above 1.50, or to measurements made at temperatures above 30°C.

TEST SUMMARY

The refractive index is measured by the critical angle method with a Bausch and Lomb Precision Refractometer using monochromatic light of an optical-mechanical or automatic digital type with the prism temperature accurately controlled. The instrument is previously adjusted by means of a solid reference standard and the observed values are corrected, when necessary, by a calibration obtained with certified liquid standards.

NOTE—*The manual instrument is out of production since 1976. The only way to obtain it may be through used equipment suppliers*.

TEST PRECISION

Procedure	Repeatability	Reproducibility
A (manual)	RI 0.0002	0.0005
	RD 0.00012	0.00012
B (Automatic)	RI 0.0002	0.0005

The bias compared against pure reference materials is not expected to be more than:

Refractive Index:	±0.00006
Refractive Dispersion:	±0.00012

REFRACTIVE INDEX OF VISCOUS MATERIALS: D 1747

EXPLANATION

Refractive index is a fundamental physical property that can be used in conjunction with other properties to characterize pure hydrocarbons and their mixtures. The use of refractive index in correlative methods for the determination of the gross composition of viscous oils and waxes often requires its measurement at elevated temperature.

Refractive index is the ratio of the velocity of light (of specified wavelength) in air, to its velocity in the substance under examination. The relative index of refraction is defined as the sine of the angle of incidence divided by the size of the angle of refraction, as light passes from air into the substance. If absolute refractive index (that is, referred to vacuum) is desired, this value should be multiplied by the factor 1.00027, the absolute refractive index of air. The numerical value of refractive index of liquids varies inversely with both wavelength and temperature.

This test method covers the measurement of refractive indexes, accurate to two units in the fourth decimal place, of transparent and light-colored viscous hydrocarbon liquids and melted solids which have refractive indexes in the range between 1.33 and 1.60, and at temperatures from 80 to 100°C. Temperatures lower than 80°C may be used provided that the melting point of the sample is at least 10°C below the test temperature.

This test method is not applicable, within the accuracy stated, to liquids having colors darker than ASTM Color No. 4. ASTM color as determined by Test Method D 1500, to liquids which smoke or vaporize readily at the test temperature, or to solids melting within 10°C of the test temperature.

The instrument can be successfully used for refractive indices above 1.60, but since certified liquid standards for ranges above 1.60 are not yet available, the accuracy of measurement under these conditions has not been evaluated.

TEST SUMMARY

The refractive index normally is measured by the critical angle method using monochromatic light from a sodium lamp. The instrument is previously adjusted by means of calibration obtained with certified liquid standards.

TEST PRECISION

Repeatability:	0.00007
Reproducibility:	0.0006

RESIDUES IN LIQUEFIED PETROLEUM GASES: D 2158

EXPLANATION

Control over the residue content (required by Specification D 1835) is of considerable importance in the end-set applications of LPG. In liquid feed systems the residues may lead to troublesome deposits and, in vapor offtake systems, residues that are carried over can foul regulating equipment. Those that will remain will accumulate, can be corrosive, and will contaminate following product. Water, particularly if alkaline, can cause failure of regulating equipment and corrosion of metals. This test covers the determination of the extraneous materials weathering above 38°C that are present in liquefied petroleum gases. LPG that contain alcohols to enhance their anti-icing behavior can give erroneous results by this test method. The results can be expressed in terms of measured volumes or indices derived from these volumes. In either case, the test method provides an indication of the quantity and nature of materials in the product that are substantially less volatile than the LPG hydrocarbons.

Although this test method has been used to verify cleanliness and lack of heavy contaminants in propane for many years, it may not be sensitive enough to protect some equipment from operational problems or increased maintenance. A more sensitive test, able to detect to lower levels of dissolved contaminants, may be required for some applications.

TEST SUMMARY

A 100 mL sample of LPG is weathered in a 100 mL centrifuge tube. The volume of residue remaining at 38°C is measured and recorded as is also the appearance of a filter paper to which the residue has been added in measured increments.

TEST PRECISION

Parameter Measured		Repeatability	Reproducibility
O Number	0–20	4	6
	20–40	6	8
	40–100	8	12
R Number	0–20	5	10
	20–40	10	20
	40–100	20	30

The procedure in this test method has no bias because the residues are defined in terms of this test method.

ROLL STABILITY OF LUBRICATING GREASES: D 1831

EXPLANATION

Although this test is widely used for specification purposes, the significance of roll stability test has not been determined. The changes in worked penetration of a grease after rolling are believed to be a measure of its shear stability, under the conditions of this low shear test. The roll stability is defined as the change in consistency of a sample after a specified amount of working in a test apparatus utilizing a weighted roller inside a rotating cylinder.

TEST SUMMARY

Test Method D 1403 cone penetration of an approximately 50 g aliquot of lubricating grease is determined. The grease is then subjected to low shear at 20 to 35°C for 2 h in a standard roll stability apparatus, before the cone penetration is again measured. The difference between the cone penetration before and after working is used as a measure of the effect of low shear working on grease consistency.

TEST PRECISION

Range of samples:	NLGI Consistency Nos. 1 and 2
Repeatability:	11 penetration units—tenths of a mm
Reproducibility:	45 penetration units—tenths of a mm

RUST PREVENTIVE CHARACTERISTICS OF ENGINE OILS: D 6557

EXPLANATION

This bench test method was designed as a replacement for Test Method D 5844 which was designed to measure the ability of an engine oil to protect valve train components against rusting or corrosion under low temperature, short trip service, and was correlated with vehicles in that type of service prior to 1978. Correlation between these two test methods has been demonstrated for most, but not all, of the test oils evaluated. This test method covers a Ball Rust Test (BRT) procedure for evaluating the anti-rust ability of fluid lubricants. The procedure is particularly suitable for the evaluation of automotive engine oils under low-temperature, acidic service conditions. ASTM Test Monitoring Center occasionally publishes Information Letters to update this test method.

TEST SUMMARY

Multiple test tubes, each containing test oil and a specimen, are placed in a test tube rack, which is attached to a mechanical shaker. The shaker speed and temperature are controlled. Air and an acidic solution are continuously fed into each test tube over an 18 h period to prevent a corrosive environment. The specimens are then removed, rinsed, and analyzed by an optical imaging system designed to quantify the antirust capability of each test oil.

TEST PRECISION

Repeatability:	15.15 AGV
Reproducibility:	18.89 AGV

No bias is believed to exist. However, this aspect of the test method will be reevaluated after the test method has been in use by several laboratories over an appropriate period of time.

RUST PREVENTING CHARACTERISTIC OF MINERAL OILS:

D 665

(Equivalent Test Methods: IP 135, ISO 7120, DIN 51585, JIS K 2510, and AFNOR T60-151)

EXPLANATION

In many instances, such as in the gears of a steam turbine, water can become mixed with the lubricant, and rusting of ferrous parts can occur. This test method indicates how well inhibited mineral oils aid in preventing this type of rusting. This test method is also used for testing hydraulic and circulating oils, including heavier-than-water fluids.

TEST SUMMARY

A mixture of 300 mL of the oil under test is stirred with 30 mL of distilled water or synthetic sea water, as required, at a temperature of 60°C(14°F) with a cylindrical steel specimen completely immersed therein. It is customary to run the test for 24 h; however, the test period may, at the discretion of the contracting parties, be for a shorter or longer period. The specimen is observed for signs of rusting and, if desired, degree of rusting.

TEST PRECISION

Since the results of the test are only intended to give a pass/fail rating to the oil being tested, no statement is made about either the precision or the bias of this test method.

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RUST PREVENTING CHARACTERISTICS OF STEAM TURBINE OIL:

D 3603

EXPLANATION

Horizontal metal surfaces, on which water droplets tend to be retained, are more prone to rusting and corrosion than vertical or sloping surfaces. This test method is therefore more discriminating than Test Method D 665 (Procedure A), since it gives a separate evaluation of the oil in a horizontal and a vertical surface. The test method indicates the ability of oils to prevent rusting and corrosion of all ferrous surfaces in steam turbines under full flow and quasi-static conditions. It is used for specification of new oils.

TEST SUMMARY

A horizontal steel disk and vertical steel cylinder are immersed in a stirred mixture of 275 mL oil and 25 mL of distilled water at a temperature of 60°C. The horizontal specimen allows water to puddle on the surface, and the vertical specimen is continually washed with the oil-water mixture during the test. The test is run for 6 h and the specimens are evaluated.

TEST PRECISION

Since this is a pass-fail test, it is not practical to specify the precision of this test method.

No statement of bias is being made.

SALTS IN CRUDE OIL
D 3230

EXPLANATION

The knowledge of the content of salt in crude oil is important in deciding whether or not the crude needs desalting. Excessive salt left in the crude frequently results in higher corrosion in refining units.

TEST SUMMARY

This test method is based on the conductivity of a solution of crude oil in a polar solvent when subjected to an alternating electrical stress. The sample is dissolved in a mixed solvent and placed in a test cell consisting of a beaker and two parallel stainless steel plates. An alternating voltage is passed through the plates and the resulting current flow is shown on a milliammeter. The salt content is obtained by reference to a calibration curve of current versus salt content of known mixtures.

TEST PRECISION

This test method has been extensively tested in a number of laboratories, and found to give results comparable to those from other procedures for determining salt in crude oil.

	mg/kg	lbs/1000 bbl
Repeatability:	0.3401 $X^{0.75}$	0.2531 $X^{0.75}$
Reproducibility:	2.7803 $X^{0.75}$	2.069 $X^{0.75}$

Where X is the average of two test results.
This test procedure has no bias.

SALT IN CRUDE OILS:
D 6470

EXPLANATION

A knowledge of water extractable inorganic halides in crude oil is important in deciding whether or not the oil needs desalting. Excessive halides, especially in crude oil, frequently results in higher corrosion rates in refining units. This test method covers the determination of salt in crude oils. For the purpose of this test method, the salt is expressed as % (m/m) NaCl, and covers the range from 0.0005–0.15 % (m/m). The detection limit of this test method is 0.0002 % (m/m) for salt as NaCl. This test method is applicable to nearly all of the heavier petroleum products, such as crude oils, residues, and fuel oils. It may also be applied to used turbine oil and marine diesel fuel to estimate seawater contamination. Water extractable salts, originating from additives present in oils, are codetermined.

TEST SUMMARY

After homogenizing the crude oil with a mixer, a weighed aliquot is dissolved in xylene at 65°C and extracted with specified volumes of alcohol, acetone, and water in an electrically heated extraction apparatus. A portion of the aqueous extract is analyzed for total halides by potentiometric titration with standard silver nitrate solution.

TEST PRECISION

Repeatability:	0.0243 $X^{0.612}$
Reproducibility:	0.0477 $X^{0.612}$

Where X is the salt concentration in mass % as NaCl.

This procedure has no bias since the salt content is defined only in terms of this test method, and certified reference materials are not available. However, based on the spiked samples used in the interlaboratory study, the following observations can be made:

Over the range of 0.0005 to 0.0400 mass % salt added, the recovery was constant and averaged 97 %. Over the range 0.0400 to 0.1500 mass % salt, the recovery was a function of concentration and gradually decreased from 97 % at 0.04 mass % to 88 % at the 0.15 mass % level.

SAPONIFICATION NUMBER: D 94

(Equivalent Test Methods: IP 136, ISO 6293, DIN 51559, JIS K 2503, and AFNOR T60-110)

EXPLANATION

Petroleum products may contain additives that react with alkali to form metal soaps. Certain used engine oils may also contain chemicals which will similarly react with alkali. The saponification number expresses the amount of base which will react with 1 g of the sample when heated in a specific manner. Since compounds of sulfur, phosphorus, halogens, and certain other elements, which are sometimes added to petroleum products also consume alkali and acids, the results obtained indicate the effect of these extraneous materials in addition to the saponifiable material present.

TEST SUMMARY

A known weight of the sample is dissolved in methylethylketone or a mixture of suitable solvents. It is heated with a known amount of standard alcoholic potassium hydroxide for between 30 to 90 min at 80°C. At the end, the excess alkali is titrated with standard hydrochloric acid and the saponification number calculated. In Test Method D 94, Method A—Color Indicator Titration, the titration is carried out manually with phenolpthalein indicator; in Test Method D 94, Method B—Potentiometric Titration method, the titration is carried out potentiometrically.

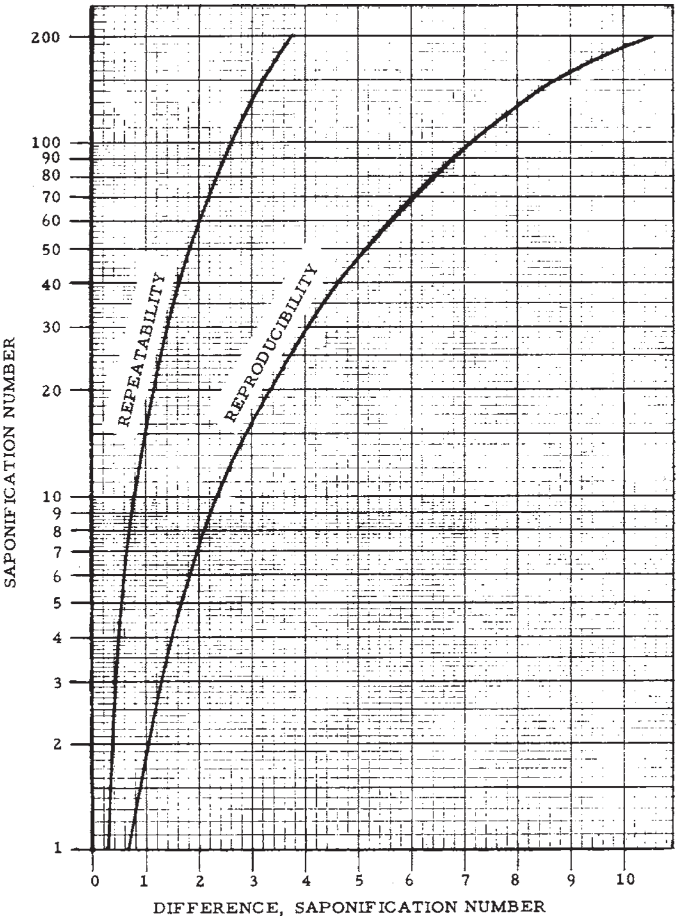

FIG. 18—*Precision Data.*

TEST PRECISION

The precision of Method A procedure is given in Fig. 18. The precision of Method B, Potentiometric Procedure is:

Repeatability:	2.76 mg KOH/g
Reproducibility:	10.4 mg KOH/g

The bias of this test method is not known.

SEDIMENT TESTS SEDIMENT IN CRUDE AND FUEL OILS:

D 473

(Equivalent Test Methods: IP 53, ISO 3735, DIN 51789, and AFNOR M07-063)

GENERAL

There are several sediment tests that differ from each other only in details of the procedure such as the solvent used, time, or speed of centrifugation, or both.

EXPLANATION

A knowledge of the sediment content of crude and fuel oils is important both to the refining operations and the buying or selling of the oil.

TEST SUMMARY

An oil sample contained in a refractory thimble is extracted with hot toluene until the residue reaches a constant mass.

TEST PRECISION

In the sediment range 0 to 0.4 % sediment, the repeatability is $0.017+0.255\ X$, and reproducibility is $0.033+0.255\ X$, where X is the average sediment result in percent. Precision on recycled oils and crank case oils is unknown.

SEDIMENT TESTS IN CRUDE OIL:

D 4807

EXPLANATION

See Test Method D 473. This test method is applicable to crude oils with sediments up to about 0.15 mass %.

TEST SUMMARY

A crude oil sample is dissolved in hot toluene and filtered under vacuum through a 0.45 μm porosity membrane filter. The filter with residue is washed, dried, and weighed.

TEST PRECISION

Repeatability:	$0.04388\ X^{0.5}$
Reproducibility:	$0.1176\ X^{0.5}$

More sediment is recovered from crude oil by this test method compared to the results by Test Method D 473.

SEDIMENT TESTS IN TRACE SEDIMENT IN LUBRICATING OILS:

D 2273

EXPLANATION

Excessive amounts of sediment in oil could lead to system malfunction in critical applications. This test method measures the trace level amounts (<0.05 volume %) of naptha-insoluble sediment that can be separated by centrifugation. The test method is not applicable in cases where precipitated oil-soluble components will appreciably contribute to the sediment readings.

TEST SUMMARY

A 100-mL sample of oil is mixed with 50 mL of ASTM precipitation naptha, and is heated in a water bath at 32 to 35°C for 5 min. The centrifuge tube containing the heated mixture is centrifuged for 10 min at a rate of between 600 to 700 relative centrifuge force (rcf). After decanting the mixture carefully, the procedure is repeated with another portion of naptha and oil. The final reading of sediment is recorded.

TEST PRECISION

Sediment, percent volume	Repeatability	Reproducibility
0.000–0.002	0.001	0.001
0.003–0.005	0.001	0.001
0.006–0.01	0.002	0.002

This is an empirical test and no statement of bias can be made.

SEDIMENT TESTS TOTAL SEDIMENT IN RESIDUAL FUELS:

D 4870

(Equivalent Test Methods: IP 375 and ISO 10307)

EXPLANATION

Appreciable amounts of sediment in a residual fuel oil can foul the handling facilities, and give problems in burner mechanisms. Sediment can accumulate in storage tanks, on filter screens, or on burner parts, resulting in obstruction of the oil flow from the tank to the burner. This test method is applicable up to sediment levels of 0.40 m/m % for distillate fuel oils containing residual components, and to 0.50 m/m % in residual fuel oils having a maximum viscosity of 55 cSt at 100°C. Some fuels may exceed the maximum filtration time specified in the test due to factors other than the presence of significant quantities of insoluble organic or inorganic material.

TEST SUMMARY

A 10-g sample of oil is filtered through the prescribed apparatus at 100°C. After washing with the solvent, and drying, the total sediment on the filter medium is weighed. The test is to be carried out in duplicate.

TEST PRECISION

Sample	Repeatability	Reproducibility
Residual Fuels	0.123 X	0.341 X
Distillate Fuels	0.048 X	0.174 X

Where X is the average of the test result in m/m %.

The bias of this test method has not been determined.

SEDIMENT TESTS WATER AND SEDIMENT IN CRUDE OIL:
D 96

EXPLANATION

A determination of sediment and water content is required to determine accurately the net volume of crude oil involved in sales, taxation, exchanges, inventories, and custody transfers. An excessive amount of sediment and water in crude oil is significant because it can cause corrosion of equipment and problems in processing and transporting and may violate federal, state, or municipal regulations. This test method may not always provide the most accurate results, but it is considered the most practical method for field determination of sediment and water. For a higher degree of accuracy, Test Methods D 4006, D 4377, or D 473 should be used.

TEST SUMMARY

Known volumes of crude oil and solvent are placed in a centrifuge tube and heated to 60°C. After centrifugation, the volume of the sediment-and-water layer at the bottom of the tube is read. For some waxy crude oils, temperatures of 71°C or higher may be required to completely melt the wax crystals so that they are not measured as sediment.

TEST PRECISION

The precision or bias of this test method has not yet been determined.

SEDIMENT TESTS WATER AND SEDIMENT IN MIDDLE DISTILLATE FUELS:
D 2709

EXPLANATION

Appreciable amounts of water and sediment in a fuel oil tend to cause fouling of the fuel handling facilities and to give trouble in the fuel system of a burner or engine. An accumulation of sediment in storage tanks and on filter screens can obstruct the flow of oil from the tank to the combustor. Water in middle distillate fuels can cause corrosion of tanks and equipment, and if detergent is present, the water can cause emulsions or a hazy appearance. Water is necessary to support microbiological growth at fuel-water interfaces in fuel systems.

This test method is used as an indication of water and sediment in middle distillate fuels having viscosities at 40°C in the range of 1.0 to 4.1 cSt and densities in the range of 770 to 900 kg/m^3.

TEST SUMMARY

A sample of fuel is centrifuged at an rcf of 800 for 10 min at 21 to 32°C in a centrifuge tube readable to 0.005 mL and measurable to 0.01 mL. After centrifugation, the volume of water and sediment that has settled into the tip of the centrifuge tube is read to the nearest 0.005 mL.

TEST PRECISION

Repeatability:	0.014 volume percent
Reproducibility:	0.041 volume percent

The bias of this test method is not known.

LUBRICITY OF DIESEL FUELS BY SLBOCLE:
D 6078

EXPLANATION

Diesel fuel injection equipment has some reliance on lubricating properties of diesel fuel. Shortened life of engine components, such as diesel fuel injection pumps and injectors, has sometimes been ascribed to lack of lubricity in a diesel fuel. The trend of Scuffing Load Ball-on-Cylinder Lubricity Evaluator (SLBOCLE) test results to diesel injection system pump component distress due to wear has been demonstrated in pump rig tests for some fuel/hardware combinations where boundary lubrication is believed to be a factor in the operation of the component. The tangential friction force, as measured in the SLBOCLE test, is sensitive to contamination of the fluids and test materials, the presence of oxygen and water in the atmosphere, and the temperature of the test. Lubricity evaluations are also sensitive to trace contaminants acquired during test fuel sampling and storage.

This test method is applicable to middle distillate fuels, such as Grades Low Sulfur No. 1D and 2D, No. 1D and 2D diesel fuels, in accordance with Specification D 975, and other similar petroleum-based fuels which can be used in diesel engines. It is not known that this test method will predict the performance of all additive/fuel combinations. Additional work is underway to further establish this correlation. The SLBOCLE and High Frequency Reciprocating Rig (HFRR) Test Method D 6079 are two methods for evaluating diesel fuel lubricity. No absolute correlation has been developed between these two test methods. The SLBOCLE may be used to evaluate the relative effectiveness of diesel fuels for preventing wear under the prescribed test conditions. If a standard SLBOCLE rating has been set, then the single load test provides a more rapid evaluation than the incremental load test. Correlation of SLBOCLE test results with field performance of diesel fuel injection systems has not yet been determined. This test method is designed to evaluate boundary lubricating properties. While viscosity effects on lubricity in this test method are not totally eliminated, they are minimized.

TEST SUMMARY

A 50 mL test specimen of fuel is placed in the test reservoir of an SLBOCLE and adjusted to the test temperature of 25°C. When the temperature has stabilized, 50 % relative humidity air is used to aerate the fuel at 0.5 L/min while 3.3 L/min flows over the fuel for 15 min. During the remainder of the test sequence, 50 % relative humidity air flows over the fuel at a rate of 3.8 L/min. A load arm holding a nonrotating steel ball and loaded with a 500 g mass is lowered until it contacts a partially fuel immersed polished steel test ring rotating at 525 r/min. The ball is caused to rub against the test ring for a 30 s break in period before beginning an incremental-load or a single-load test. Wear tests are conducted by maintaining the ball in contact with the partially immersed 525-r/min test ring for 60 s. For incremental load tests, the test ring is moved at least 0.75 mm for each new load prior to bringing a new ball into contact with the test ring. The tangential friction force is recorded while the ball is in contact with the test ring. The friction coefficient is calculated from the tangential friction force. In the incremental-load test, the minimum applied load required to produce a friction coefficient greater than 0.175 is an evaluation of the lubricating properties of the diesel fuel. In the single-load test, a friction coefficient less than or equal to 0.175 indicated the diesel fuel passes the lubricity evaluation, while a friction coefficient greater than 0.175 indicated the diesel fuel fails the lubricity evaluation.

TEST PRECISION

Repeatability:	900 g
Reproducibility:	1500 g

The procedure in this test method has no bias because lubricity is not a fundamental and measurable fluid property, and thus is evaluated in terms of this test method.

SONIC SHEAR STABILITY SHEAR STABILITY INDEX: D 3945

EXPLANATION

These test methods (Kurt Orbahn) measure the percent viscosity loss at 100°C of polymer-containing fluids when evaluated by either of two diesel injector apparatus procedures. Procedure A uses European Diesel Injector Test Equipment and Procedure B uses fuel injector shear stability test (FISST) equipment. The viscosity loss reflects polymer degradation due to shear at the nozzle.

Both Procedure A (using European Diesel Injector Test Equipment) and Procedure B (using FISST equipment) of this test method evaluate the percent viscosity loss for polymer-containing fluids resulting from polymer degradation in the high shear nozzle device. Minimum interference from thermal or oxidative effects would be anticipated. The two procedures exhibit essentially equal percent viscosity loss for each oil used in developing this test method. Both procedures also show essentially comparable repeatability and reproducibility.

These test methods are not intended to predict viscosity loss in field service for different polymer classes or for different field equipment. However, it may be possible to establish some correlation for a specific polymer type in specific field equipment.

TEST SUMMARY

The polymer-containing fluid is passed through a diesel injector nozzle at a shear rate that causes the less shear stable polymer molecules to degrade. The resultant degradation reduces the kinematic viscosity of the fluid under test.

The reduction in kinematic viscosity, reported as percent loss of the initial kinematic viscosity, is a measure of the shear stability of the polymer-containing fluid.

TEST PRECISION

	European Diesel Injector Test	Fuel Injector Shear Stability Test
Repeatability	1.43 %	1.19 %
Reproducibility	4.57 %	5.22 %

SONIC SHEAR STABILITY OF POLYMER-CONTAINING FLUIDS:
D 6278

EXPLANATION

This test method evaluates the percent viscosity loss for polymer-containing fluids resulting from polymer degradation in the high shear nozzle device. Thermal or oxidative effects are minimized. This test method is not intended to predict viscosity loss in field service in different field equipment under widely varying operating conditions, which may cause lubricant viscosity to change due to thermal and oxidative changes as well as by the mechanical shearing of the polymer.

Test Method D 2603 has been used for similar evaluation of shear stability, but no detailed attempt has been made to correlate the results by two methods. Test Method D 5275 also shears oils in a diesel injector apparatus, but may give different results. This test method has different calibration and operational requirements than Test Method D 3945. This test method uses test apparatus as defined in CEC L-14-A-93 Test Method, but differs in the period of time required for calibration.

TEST SUMMARY

The test method measures the percent viscosity loss at 100°C of polymer-containing fluids when evaluated by a diesel injector apparatus procedure that uses European diesel injector test equipment. The fluid is passed through a diesel injector nozzle at a shear rate that causes polymer molecules to degrade. The resultant degradation reduces the kinematic viscosity of the fluid under test. The percent viscosity loss is a measure of the mechanical shear stability of the polymer-containing fluid.

TEST PRECISION

Repeatability:	1.05 %
Reproducibility:	2.68 %

No estimate of bias for this test method can be justified.

SONIC SHEAR STABILITY OF HYDRAULIC FLUID:
D 5621

EXPLANATION

This test method permits the evaluation of shear stability with minimum interference from thermal and oxidative factors that may be present in some applications. It is applicable to fluids containing both readily sheared and shear-resistant polymers. Correlation with performance in the case of hydraulic application has been established.

TEST SUMMARY

Hydraulic fluid is irradiated in a sonic oscillator for a period of time and the change in viscosity is determined by Test Method D 445. A standard reference fluid containing a readily sheared polymer is run frequently to ensure that the equipment imparts a controlled amount of sonic energy to the sample.

TEST PRECISION

Repeatability:	0.38 cSt
Reproducibility:	0.60 cSt

The bias of this test method has not been determined.

SONIC SHEAR STABILITY OF POLYMER-CONTAINING OILS:
D 2603

EXPLANATION

This test method covers the evaluation of the shear stability of an oil sample containing polymer in terms of the permanent loss in viscosity that results from irradiating an oil sample in a sonic oscillator. This test method can be useful in predicting the continuity of this property in an oil where no change is made in the base stock or the polymer. It is not intended to predict the performance of the polymer-containing oils in service. This test method has been successfully applied to hydraulic fluids, transmission fluids, tractor fluids, and other fluids of similar applications. It is applicable to both readily sheared and shear-resistant polymers. Correlation with performance in the case of automotive engine applications has not been established.

Some of the parts for this test may not be available; Test Methods D 6278 and D 5275 are alternative tests for this work.

TEST SUMMARY

Polymer-containing oil is irradiated in a sonic oscillator for a period of time and the change in viscosity of the oil is determined by Test Method D 445. Standard reference fluids containing either a readily sheared or shear-resistant polymer are run frequently to ensure that the equipment imparts a controlled amount of sonic energy to the sample.

TEST PRECISION

Repeatability:	1.6 %
Reproducibility:	3.3 %

This test method has no bias.

SHEAR STABILITY OF POLYMER CONTAINING FLUIDS USING A EUROPEAN DIESEL INJECTOR APPARATUS:
D 7109

EXPLANATION

This test method evaluates the percent viscosity loss of fluids resulting from physical degradation in the high shear nozzle device. Thermal or oxidative effects are minimized. This test method may be used by manufacturers of polymeric lubricant additives and their customers. This test method is not intended to predict viscosity loss in field service in different field equipment under widely varying operating conditions, which may cause lubricant viscosity to change due to thermal and oxidative changes, as well as by the mechanical shearing of polymer. However, when the field service condition, primarily or exclusively, result in the degradation of polymer by mechanical shearing, there may be a correlation between the results from this test method and field results.

This test method measures the viscosity loss, in mm^2/s and percent, at 100°C of polymer-containing fluids when evaluated by a diesel injector apparatus procedure that uses European diesel injector test equipment. The viscosity loss reflects polymer degradation due to shear at the nozzle. Viscosity loss is evaluated after both 30 and 90 cycles of shearing. In general, there is no correlation between results after these two cycles of shearing. Test Method D 6278 uses essentially the same procedure with 30 cycles instead of both 30 and 90 cycles. The correlation between results from this test method at 30 cycles and results from Test Method D 6278 has not been established. Test Method D 2603 has been used for similar evaluation of shear stability. No detailed attempt has been made to correlate the results of this test method with those of the sonic shear method. This test method uses test apparatus as defined in CEC L-14-A-93; however, they differ in the period of time required for calibration. Test Method D 5275 also shears oils in a diesel injector apparatus, but may give different results. This test method has different calibration and operational requirements than Test Method D 3945.

TEST SUMMARY

A polymer containing fluid is passed through a diesel injector nozzle at a shear rate that may reduce its kinematic viscosity. The percent viscosity loss is a measure of the mechanical shear stability of the fluid. This test method may also be used for oils not containing polymer. It might not be known whether an oil submitted for test contains a polymer.

TEST PRECISION

Based on limited interlaboratory test results following preliminary precision was found. Further more extensive study is underway.

	PVL_{30}	PVL_{90}
Repeatability	1.4 %	1.8 %
Reproducibility	2.5 %	2.9 %

All test results are relative to those of the calibration fluid. Therefore no estimate of bias can be justified.

SLUDGING AND CORROSION TENDENCIES OF INHIBITED MINERAL OILS:

D 4310

EXPLANATION

Insoluble material may form in oils subjected to oxidizing conditions. This test method is used to evaluate the tendency of inhibited mineral oil based steam turbine lubricants and mineral oil based anti-wear hydraulic oils to corrode copper catalyst metal and to form sludge during oxidation in the presence of oxygen, water, and copper and iron metals at an elevated temperature. The test method is also used for testing circulating oils having a specific gravity less than that of water and containing rust and oxidation inhibitors. Significant formation of oil insolubles or metal corrosion products, or both, during this test may indicate that the oil will form insolubles or corrode metals, or both, during field service. However, no correlation with field service has been established.

This test method is a modification of Test Method D 943 where the oxidation stability of the same kind of oils is determined by following the acid number of oil.

TEST SUMMARY

An oil sample is contacted with oxygen in the presence of water and iron-copper catalyst at 95°C for 100 h. The weight of the insoluble material is determined gravimetrically by filtration of the oxidation tube contents through a 5-μm pore size filter disk. The total amount of copper in the oil, water, and sludge phases is also determined.

TEST PRECISION

Analysis	Repeatability	Reproducibility
Insoluble Material	$4.6\ X^{0.66}$	$6.3\ X^{0.66}$
Total Copper Loss	$1.2\ X^{0.80}$	$3.3\ X^{0.80}$

This precision statement is based on the samples in the range of 4.6 to 250 mg insoluble material, and 0.9 to 30 mg total copper.

This procedure has no bias.

SMOKE POINT OF KEROSENE AND AVIATION TURBINE FUEL:

D 1322

(Equivalent Test Methods: IP 57, ISO 3014, DIN 51406, JIS K 2537, and AFNOR M07-028)

EXPLANATION

The smoke point (and Luminometer number with which it can be correlated) is quantitatively related to the potential radiant heat transfer from the combustion products of the fuel. Because radiant heat transfer exerts a strong influence on the metal temperature of combustor liners and other hot sections of gas turbines, the smoke point provides a basis for correlation of fuel characteristics with the life of these components.

This test method provides an indication of the relative smoke producing properties of kerosene and aviation turbine fuels in a diffusion flame. The smoke point is related to the hydrocarbon type composition of such fuels. Generally the more aromatic the fuel, the smokier the flame. A high smoke point number indicates a fuel of low smoke producing tendency.

TEST SUMMARY

The sample is burned in an enclosed wick-fed lamp that is calibrated daily against pure hydrocarbon blends of known smoke point. The maximum height of flame that can be achieved with the test fuel without smoking is determined to the nearest 0.5 mm.

TEST PRECISION

Repeatability:	2 mm
Reproducibility:	3 mm

This test method has no bias.

SOFTENING POINT OF ASPHALT AND PITCH:

GENERAL

Asphalt and pitch do not go through a solid-liquid phase change when heated, and therefore do not have true melting points. As the temperature is raised, they gradually soften or become less viscous. For this reason, the determination of the softening point must be made by an arbitrary but closely defined method, if the test values are to be reproducible.

Softening point determination is useful in determining the consistency as one element in establishing the uniformity of shipments or sources of supply.

There are four test methods for this analysis, which are described here in brief.

SOFTENING POINT METTLER CUP-AND-BALL METHOD: D 3461

TEST SUMMARY

This test method is applicable in the temperature range 50 to 180°C, and gives results comparable to those obtained by the Test Method D 36. The sample is suspended in a cylindrical cup with a 6.5-mm hole in the bottom and with a lead ball, 8 mm in diameter, centered on top of the sample in the cup, flows downward a distance of 19 mm to interrupt a light beam, as the sample is heated at a linear rate in air.

TEST PRECISION

Repeatability:	0.5°C
Reproducibility:	1.5°C

This test method has no bias.

SOFTENING POINT CUBE-IN-WATER METHOD: D 61

TEST SUMMARY

This test method covers the determination of the softening point of pitches below 176°F(80°C). Pitches of higher softening point should be tested by Test Methods D 2319 or D 3104 (see the next pages).

Two cubes of pitch, supported on wire hooks, are heated at controlled rate in water in a glass container. The softening point is defined as the mean of the temperatures at which the cubes sag downwards a distance of 25 mm.

TEST PRECISION

Repeatability:	3°F(1.5°C)
Reproducibility:	5°F(3°C)

This test method has no bias.

SOFTENING POINT CUBE-IN-AIR METHOD: D 2319

TEST SUMMARY

This test method covers the determination of softening point above 176°F(80°C). Test Method D 3104 gives comparable results.

Two cubes of pitch, supported on wire hooks, are heated in a standardized air oven at a linear rate. The softening point is the mean of the temperatures at which the cubes sag downwards a distance of 60 mm.

TEST PRECISION

Repeatability:	2°F(1.0°C)
Reproducibility:	7.2°F(4.0°C)

This test method has no bias.

SOFTENING POINT METTLER SOFTENING POINT METHOD: D 3104

TEST SUMMARY

This test method is applicable to the pitches with softening points in the range 50 to 180°C. This test method and Test Method D 2319 give comparable results.

Pitch is suspended in a cylindrical cup with a 6.35-mm hole in the bottom. As the sample is heated in air at a linear rate, the pitch flows downward a distance of 19 mm to interrupt a light beam. This is considered as the softening point in this test.

TEST PRECISION

Repeatability:	0.5°C
Reproducibility:	1.5°C

This test method has no bias.

SOLIDIFICATION POINT OF PETROLEUM WAX: D 3944

EXPLANATION

Solidification point of petroleum wax is defined as the temperature in the cooling curve of the wax where the slope of the curve first changes significantly as the wax sample changes from a liquid to a solid state. The related methods of determining the melt point of petroleum wax are relatively time-consuming. This test method reduces the test time significantly and at the same time gives a reasonable precision. This test method is also applicable to similar materials such as synthetic waxes, but the precision may vary.

TEST SUMMARY

A sample of wax is placed in a test tube at ambient temperature and heated above the solidification point of the wax sample. A thermocouple probe, attached to a recorder, is inserted into the wax sample, which is allowed to cool to room temperature. The thermocouple response of the cooling wax traces a curve on the chart paper of the recorder. The first significant change in the slope of the curve is considered as the softening point.

TEST PRECISION

Product	Repeatability	Reproducibility
Distillate Waxes	0.6°C(1.0°F)	1.2°C(2.2°F)
Residual Waxes	0.7°C(1.3°F)	2.4°C(4.3°F)

SOLVENT RED DYE 164 IN DIESEL FUELS:
D 6258

EXPLANATION

This test method was developed to provide for the enforcement of 26 CFR 48.4082-1(b), which mandates that all tax exempt diesel fuels be dyed with an amount of solvent Red 164 at a concentration that is spectrally equivalent to 11.1 mg/L of Solvent Red 26. The test is used to verify that the correct amount of dye has been added at terminals, or refineries before sale. Solvent Red 26 is considered as a reference standard because it is available in certified pure form. On the other hand, composition of solvent Red 164 dye varies but it has higher solubility and is relatively less expensive.

TEST SUMMARY

The absorbance of each sample is recorded over a specified wavelength range, and the scan is analyzed using derivative analysis software to determine the dye concentration.

TEST PRECISION

Repeatability:	$0.1847\ X^{0.5}$
Reproducibility:	$0.7652\ X^{0.5}$

Where X is the mean dye concentration in mg/L of two results.

The bias of this test method has not been determined.

STABILITY AND COMPATIBILITY OF HEAVY FUEL OILS AND CRUDE OILS BY OIL STABILITY ANALYZER (OPTICAL DETECTION):
D 7112

EXPLANATION

Automatic determination of stability parameters using a light back-scattering technique improves accuracy and removes human errors. In manual testing, operators have to visually compare the oil stains on pieces of filter paper to determine if asphaltenes have been precipitated. Refinery thermal and hydrocracking processes can be run closer to their severity limits if stability parameters can be calculated more accurately. This gives increased yield and profitability. Results from this test method could be used to set a standard specification for stability parameter for fuel oils. The compatibility parameters of crude oils can be used in crude oil blending in refineries to determine, in advance, which crude oil blends will be compatible and thus can be used to minimize plugging problems, unit shut downs, and maintenance costs. Determination of crude oil compatibility parameters also enables refineries to select crude oil mixtures more economically. This test method measures stability and compatibility parameters, and determines stability reserve on different blends for particular applications to optimize the blending, storage, and use of heavy fuel oils. Users of this test method would normally use stability and compatibility parameters to determine stability reserve of residual products, fuel blends, and crude oils. However, the interpretation of stability, stability reserve, and compatibility is heavily “use dependent,” and is beyond the scope of this test method.

This test method covers an automated procedure involving titration and optical detection of precipitated asphaltenes for determining the stability and compatibility parameters of refinery residual streams, residual fuel oils, and crude oils. Stability in this context is the ability to maintain asphaltenes in a peptized or dissolved state and not undergo flocculation or precipitation. Similarly, compatibility relates to the property of mixing two or more oils without precipitation or flocculation of asphaltenes. This test method is applicable to residual products from atmospheric and vacuum distillation, from thermal catalytic, and hydrocracking processes, to products typical of Specifications D 396 Grades No. 5L, 5H, and 6, and D 2880, Grades No. 3-GT and 4-GT, and to crude oils, providing these products contain 0.05 m% or greater concentration of asphaltenes. It would be pointless to apply this test method to unstable oils that already contain flocculated asphaltenes.

TEST METHOD SUMMARY

Stability and compatibility parameters are determined by titration and optical detection of precipitated asphaltenes. A stock solution is prepared and three different mixtures of the sample oil plus xylene are titrated with *n*-heptane to cause precipitation of asphaltenes. The titrated mixture is continuously circulated through an optical detector which detects precipitated asphaltenes by back-scattering of visible light. The amount of oil, xylene, and *n*-heptane are used to calculate stability parameters: solvent equivalent, P-value, and $FR_{5/1}$. If the density of a crude oil sample is known, then the compatibility parameters (S_{BN} and I_N) of the crude oil may also be calculated.

INTERFERENCES—Free water present in the oil can cause difficulties with the optical detector and should be removed by centrifuging prior to testing. Solid particles such as coke or wax particles, mud, sand, or catalyst fines in the oil will not affect the optical detector or interfere with the results.

TEST PRECISION

Only limited amount of in-house data from three laboratories are published in the test method. A full interlaboratory study will be undertaken to determine the true precision. This test method has no bias because the results of the test are defined only in terms of this test method.

STABILITY, STORAGE DISTILLATE FUEL STORAGE STABILITY AT 43°C:

D 4625

EXPLANATION

Fuel oxidation and other degradative reactions leading to sediment (and color) formation are mildly accelerated by the test conditions, compared to typical storage conditions. These test results have been shown to predict storage stability more reliably than other more accelerated tests. This test method is applicable to distillate fuels with flash points above 38°C (100°F) and 90 % distillation points below 340°C(644°F). Because of the long storage periods involved (4 to 24 weeks), this test is not suitable for quality control testing, but does provide a tool for research on storage properties of fuels. Because the environmental effects and the materials and nature of tank construction affect storage stability, the results of this test method are not necessarily the same as those obtained during a specific field storage situation.

TEST SUMMARY

Four-hundred mL of filtered fuel are stored in a borosilicate glass container at 43°C for periods of 0, 4, 8, 12, 18, and 24 weeks. After aging for a selected time period, a sample is removed from the storage, cooled to room temperature, and analyzed for filterable insolubles and for adherent insolubles.

TEST PRECISION

Repeatability:	0.62 X
Reproducibility:	2.20 X

Where X is the average of two results in mg/100 mL.

Bias statement is not applicable because of the nature of this test.

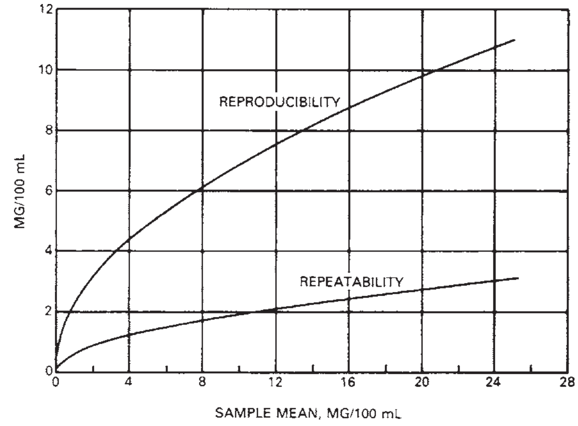

FIG. 19—*Repeatability and reproducibility for total insolubles measurements.*

STABILITY, STORAGE DISTILLATE FUEL STORAGE STABILITY:
D 5304

EXPLANATION

This test method covers a procedure for assessing the potential storage stability of middle distillate fuels, both freshly refined or already in storage, and those with or without stabilizer additives. The results of this test method are useful in comparing fuels when tested under identical conditions. The formation of insolubles is affected by the material present in the storage container and by the ambient conditions. Since this test method is conducted in glass under standardized conditions, the results from different fuels can be compared on a common basis.

TEST SUMMARY

A 100-mL aliquot of the filtered fuel is placed in a borosilicate glass container. This is placed in a pressure vessel, preheated to 90°C. The vessel is pressurized with oxygen to 800 kPa for the duration of the test. The vessel is placed in a forced air oven at 90°C for 16 h. At the end of this period, after cooling, the total amount of fuel insoluble products is determined gravimetrically and corrected for blank.

TEST PRECISION

Repeatability:	0.21 *X*
Reproducibility:	0.56 *X*

Where *X* is the average of two results in mg/100 mL.

A bias statement for this test cannot be written because of the nature of this test.

INTRINSIC STABILITY OF ASPHALTENE CONTAINING OILS:
D 7157

EXPLANATION

This test method describes a sensitive method for estimating the intrinsic stability of an oil. The intrinsic stability is expressed as an S-value. An oil with a low S-value is likely to undergo flocculation of asphaltenes when stressed (for example, extended heated storage) or blended with a range of other oils. Two oils each with a high S-value are likely to maintain asphaltenes in a peptized state and not lead to asphaltene flocculation when blended together. This test method can be used by petroleum refiners to control and optimize the refinery processes and by blenders and marketers to assess the intrinsic stability of blended asphaltene containing heavy oils. This test method is applicable to residual products from thermal and hydrocracking processes, to products typical of Specifications D 396 Grades No. 5L, 5H, and 6, and D 2880 Grades No. 3-GT and 4-GT, and to crude oils, providing these products contain 0.5 m% or greater concentrations of asphaltenes (see Test Method D 6560). This test method quantifies asphaltene stability in terms of state of peptization of the asphaltenes (S-value), intrinsic stability of the oil medium (S_o) and the solvency requirements of the peptized asphaltenes (S_a).

TEST SUMMARY

This test method uses an integrated analytical measurement system with an optical probe for the detection of asphaltene precipitation from a toluene solution of the sample. Three test specimens are dissolved in three different quantities of toluene. The three specimen/toluene solutions are automatically and simultaneously titrated with *n*-heptane to cause precipitation of the asphaltenes. The optical probe monitors the formation of flocculated asphaltenes during the titration. Flocculated asphaltenes will alter the detected light intensity. Start of flocculation is interpreted when the optical probe detects a significant and sustained decrease in rate-of-change of the light intensity. A computer routinely calculates the stability parameters and subsequently the intrinsic stability of the oil from the added *n*-heptane at the inversion point, the mass of specimen, and the volume of toluene, for the three specimen/toluene solutions.

INTERFERENCES—High content of insoluble inorganic matter (sediment) will cause some interference in this test method. In this case, the insoluble matter shall be removed by filtration according to Test Method D 4870. Also, free water in the oil can cause difficulties with the optical detector and should be removed by any suitable means (for example, centrifugation) prior to testing.

TEST PRECISION

Only preliminary precision data are available at the moment based on one laboratory. Further data will be collected through a round robin to obtain proper precision estimates.

Repeatability: for S 0.19; for S_a 0.031; and for S_o 0.12.

This test method has no bias because the results of the test are defined only in terms of this test method.

STABILITY, STORAGE STABILITY OF RESIDUAL FUELS BY SPOT TEST:

D 4740

EXPLANATION

This test method describes separate procedures for determining the cleanliness of residual fuel oil and its compatibility with a blend stock. It is applicable to fuel oils with viscosities up to 50 cSt at 100°C. These procedures used alone or together are used to identify fuels or blends that could result in excessive centrifuge loading, strainer plugging, tank sludge formation, or similar operating problems.

TEST SUMMARY

In the cleanliness procedure, a drop of the preheated sample is put on a test paper and placed in an oven at 100°C. After 1 h, the test paper is removed from the oven and the resultant spot is examined for evidence of suspended solids and rated for cleanliness using the Adjunct Reference Spots in Test Method D 4740.

In the compatibility procedure, a blend composed of equal volumes of the fuel oil sample and the blend stock is tested and rated in the same way as just described.

TEST PRECISION

Repeatability:	1 rating number
Reproducibility:	1 rating number

The bias of this test method has not been determined.

STABILITY, STORAGE STORAGE STABILITY OF WATER-IN-OIL EMULSIONS:

D 3707

EXPLANATION

This test method indicates the stability of the emulsions during storage and normal use.

TEST SUMMARY

A 100-mL sample in a graduated 100-mL cylinder is placed in a thermostatically controlled oven at 85°C for 48 or 96 h. The sample is then examined for the amount of free oil and free water separated. Additionally, water contents of the sample at specified levels in the upper and lower layers of the sample are also obtained.

TEST PRECISION

Analysis	Repeatability	Reproducibility
Free Oil, %	1	3
Free Water, %	1	1
Difference in % water content between upper and lower layer	10	14

This test method has no bias.

STABILITY, STORAGE STABILITY OF WATER-IN-OIL EMULSIONS:
D 3709

EXPLANATION

This test method indicates the ability of the emulsion to withstand mild to moderately severe winter conditions of use and storage. Generally this significance would be limited to conditions where the emulsion reaches a minimum temperature of −18°C(0°F).

TEST SUMMARY

A 100-mL sample in a graduated 100-mL cylinder is placed in a cooling box at −18°C for 16 h and then allowed to stand at room temperature for 8 h. This procedure is repeated for a total of nine times, except the fifth cycle is one of 64 h at −18°C, 8 h at room temperature. At the completion of this cycle, the sample is examined for the amount of free oil and free water separated. In addition, water contents of the sample at specified levels in the upper and lower layers of the sample are also obtained.

TEST PRECISION

Analysis	Repeatability	Reproducibility
Free Oil, %	1	2
Free Water, %	1	2
Difference in % water between upper and lower layers	3	3

This test method has no bias.

TOTAL INORGANIC SULFATE IN ETHANOL BY POTENTIOMETRIC TITRATION:
D 7318

EXPLANATION

Ethanol is used as a blending agent added to gasoline. Sulfates are indicated in filter plugging deposits and fuel injector deposits. When fuel ethanol is burned, sulfates may contribute to sulfuric acid emissions. Ethanol acceptability for use depends on its sulfate content. This method is applicable to hydrous, anhydrous ethanol, and anhydrous denatured ethanol, which is added as a blending agent with spark ignition fuels. The range of determination is 1.0 to 20 mg/kg of total inorganic sulfate.

TEST SUMMARY

An ethanol sample is titrated in ethanolic medium with a standard lead nitrate solution. Lead sulfate precipitate is formed during the titration. Perchloric acid is added to remove possible interference from carbonate. The end point is signaled by an increase in lead ion activity as measured by a lead-selective electrode.

TEST PRECISION

Repeatability	$0.1711\,(X+0.1301)^{0.2678}$
Reproducibility	$0.5807\,(X+0.1301)^{0.2678}$

where X is the results of two determinations.

Since there are no generally accepted reference materials for this property, bias of this method cannot be determined.

TOTAL AND POTENTIAL SULFATE AND INORGANIC CHLORIDE IN FUEL ETHANOL BY DIRECT INJECTION SUPPRESSED ION CHROMATOGRAPHY: D 7319

EXPLANATION

Sulfates and chlorides can be found in filter plugging deposits and fuel injector deposits. The acceptability for use of the fuel components and the finished fuels depends on the sulfate and chloride content. The method is applicable to hydrous and anhydrous denatured ethanol to be used in motor fuel applications. It is intended for the analysis of ethanol samples containing between 1.0 and 20 mg/kg of total or potential inorganic sulfate and 1.0 to 50 mg/kg of inorganic chloride.

TEST METHOD SUMMARY

For the total sulfate and chloride, a small volume of an ethanol sample is directly injected into a suitable configured ion chromatograph in accordance with manufacturer's recommendations for this test method. For potential sulfate, 0.5 mL of 30% hydrogen peroxide solution is added to 9.5 mL of the ethanol sample, and then injected into the ion chromatograph. Ions are separated based on their affinity for exchange sites of the resin with respect to the resin's affinity for the eluent. The suppressor increases the sensitivity of the test method by both increasing the conductivity of the analyte and decreasing the conductivity of the eluent. It also converts the eluent and analytes to corresponding hydrogen forms of anions. Anions are quantified by integrating their responses compared with an external calibration curve. The calibration standards are prepared in an aqueous matrix.

Interferences—Interferences can be caused by substances with similar ion chromatographic retention times, especially if they are in high concentration compared to the analyte of interest. Sample dilution can be used to minimize or resolve most interference problems.

A water dip (system void or negative peak) can cause interference with some integrators. Usually for chloride and sulfate determinations, water dip should not be a problem since their peaks are far away from the water dip.

Given the trace amounts of chloride and sulfate determined in this test method, interferences can be caused by contamination of glassware, eluent, reagents, etc. Hence, great care should be kept to ensure that the contamination is kept to a minimum.

TEST METHOD PRECISION[1]

[1]This table is Table 3 of 7319-07.

Analyte	mg/kg	Repeatability (*r*) Tri-Chamber	Reproducibility (*R*) Tri-Chamber	Repeatability (*r*) Continous	Reproducibility (*R*) Continuous
Total Chloride	1.0	0.07	0.42	0.07	0.42
	20.0	0.56	3.40	0.56	3.40
	50.0	1.06	6.45	1.06	6.45
Total Sulfate	1.0	0.09	1.22	0.25	1.60
	4.0	0.20	2.86	0.61	3.99
	20.0	0.55	7.73	1.77	11.54
Potential Sulfate	1.0	0.13	0.97	0.27	1.43
	4.0	0.33	2.55	0.71	3.73
	20.0	1.01	7.80	2.16	11.32

TOTAL AND POTENTIAL INORGANIC SULFATE AND TOTAL INORGANIC CHLORIDE IN FUEL ETHANOL BY ION CHROMATOGRAPHY USING AQUEOUS SAMPLE INJECTION:

D 7328

EXPLANATION

See explanation under D 7319 test method above.

This method can measure between 0.55 and 20 mg/kg of total inorganic sulfate, 4.0 to 20 mg/kg of potential inorganic sulfate, and 0.75 to 50 mg/kg of total inorganic chloride.

TEST SUMMARY

For total inorganic sulfate and chloride, a small volume of a sample is evaporated to dryness and reconstituted to the initial sample volume with deionized water, and injected into an ion chromatograph consisting of appropriate ion exchange columns, suppressor and a conductivity detector. For potential sulfate, the dried residue is reconstituted with 0.90% hydrogen peroxide solution in water, and injected into an ion chromatograph.

Similar methods for chloride and sulfate determinations can be found in Test Method D 5827 for engine coolant and for ethanol in ISO/CEN 15492.

Interferences—See under Test Method D 7319 above.

TEST PRECISION

Analysis	Repeatability	Reproducibility
Total Chloride	$0.02078\,(X+10.0709)$	$0.1173\,(X+10.0709)$
Total Sulfate	$0.2319\,(X+1.00^{-04})^{0.5000}$	$1.0963\,(X+1.00^{-04})^{0.5000}$
Potential Sulfate	$1.1763\,X^{0.4000}$	$1.0505\,X^{0.4000}$

where X is the average of two determinations.

Since no generally accepted reference materials are available for this analysis, no statement regarding the bias of this test method can be made.

SULFONATES BY LIQUID CHROMATOGRAPHY:

D 3712

(Equivalent Test Method: IP 369)

EXPLANATION

This test method provides a means of determining sulfonate content and of classifying and characterizing natural and synthetic petroleum sulfonate products by sulfonate content and average molecular weight. Purity of sodium sulfonate products is measured by basicity and inorganic salt contents and the reserve alkalinity of alkaline earth sulfonates by the total base number. This test method covers the analysis of refined and crude natural and synthetic oil-soluble sulfonate products. Resins, if present, are recovered with the oil phase and carboxylates are recovered as sulfonates. This test method covers the determination of mineral oil, sulfonate, water, total base number, average molecular weight, and specific gravity of calcium, barium, magnesium, sodium, and ammonium sulfonate products.

TEST SUMMARY

The sample, except a sodium sulfonate product, is dissolved in ethyl ether and converted to sulfonic acid, using dilute hydrochloric acid. The sulfonic acid after extraction is converted to sodium sulfonate and the isolated sodium sulfonate and mineral oil are dissolved in chloroform. An aliquot of the chloroform solution, or a sample of a sodium sulfonate product, dissolved in chloroform, is placed on a silica gel column. The oil is eluted with chloroform, the sulfonate with ethyl alcohol, and both are determined gravimetrically. Average molecular weight is calculated from the average equivalent weight of the sodium sulfonate, which is determined by ashing a portion of the isolated sodium sulfonate.

Water is determined by Test Method D 95. Total base number is determined by Test Method D 2896. Specific gravity is determined by pycnometer.

TEST PRECISION

Analysis	Repeatability	Reproducibility
Sulfonate, Weight %	0.63	0.92
Mineral Oil, Weight %	0.92	1.74
Water, Weight %	0.1	0.34
Specific Gravity	0.001	0.0075
Average Molecular Weight	0.000032 S^2 or 0.000032 T^2	0.000067 S^2 or 0.000067 T^2

SULFUR DETERMINATION IN PETROLEUM PRODUCTS:

GENERAL

Sulfur is present in one chemical form or other in many petroleum products. It occurs in natural form starting with crude oils, but is also added in chemical form in several products to modify performance. In some cases the presence of sulfur is beneficial to the product, and in other cases it is detrimental to the processing or use of the product. Traces of sulfur can act as catalytic poisons during processing. Particularly of concern are the sulfur oxide emissions during combustion of sulfur containing products. A number of government regulations mandate gradual elimination of sulfur from gasoline type products to curb pollution.

There are over a dozen test methods for the determination of sulfur employing a variety of techniques and applicable from trace levels to major amounts of concentration. See Table 11 for a comparison of these methods. These are described in brief. Additionally, other multielement methods such as ICPAES (Test Methods D 4951 and D 5185) and XRF (Test Methods D 4927 and D 6443) standards also determine sulfur.

SULFUR DETERMINATION BY BOMB METHOD: **D 129**
(Equivalent Test Methods: IP 61, DIN 51577, and AFNOR T60-109)

This test method is applicable to any petroleum product sufficiently low in volatility that it can be weighed accurately in an open sample boat and containing at least 0.1 % sulfur. This test method is not applicable to samples containing elements that give residues, other than barium sulfate, which are insoluble in dilute hydrochloric acid and would interfere in the precipitation step. These interfering elements include iron, aluminum, calcium, silicon, and lead, which are sometimes present in greases and lube additives. Other insoluble materials that interfere are silica, molybdenum disulfide, asbestos, mica, etc. The test method is not applicable to used oils containing wear metals, and lead or silicates from contamination. The samples that are excluded here can be analyzed by Test Method D 1552 (see this section).

TEST SUMMARY

The sample is oxidized by combustion in a bomb containing oxygen under pressure. The sulfur in the sample is converted to sulfate, and from the bomb washings is gravimetrically determined as barium sulfate.

TEST PRECISION

Sulfur, Weight %	Repeatability	Reproducibility
0.1 to 0.5	0.04	0.05
0.5 to 1.0	0.06	0.09
1.0 to 1.5	0.08	0.15
1.5 to 2.0	0.12	0.25
2.0 to 5.0	0.18	0.27

Results from one single laboratory on NIST SRMs were found to be 0.05 mass % higher than the accepted reference values.

TABLE 11—*Test Methods for Sulfur Determination in Petroleum Products.*

Test Method	Technique	Scope	Limitation	Repeatability	Reproducibility	Bias
D 129	Bomb Combustion and $BaSO_4$ precipitation	Petroleum products, lube oils, adpacks, greases >0.1% sulfur	Not applicable to samples that give insoluble residues; Fe, Al, Ca, Si, Pb; Silica, MoS_2, asbestos, mica, used oils	Dependent on level	Dependent on level	0.05 % high
D 1266	Lamp combustion: $BaSO_4$ precipitation, or NaOH titration, or turbidometry if <0.01%	0.01–0.04 % sulfur in gasoline, kerosene, naptha	Acid or base forming compounds in titration. Labor intensive	0.005	$0.10+(0.025X)$	Not known
D 1552	High temperature combustion: iodate titration or IR detection	Samples with boiling points >177°C and >0.06 % S; Petroleum cokes up to 8 % S.	*Iodate*–>1 % chlorine and >0.1 % nitrogen interfere IR–None from N or S.	Dependent on sulfur level	Dependent on level	Not known
D 2622	Wavelength dispersive XRF	Diesel and jet fuels, kerosene, distillate oils, naptha, residual oils, lube base oil, hydraulic oil, crude oil, unleaded gasoline, M-85 and M-100 fuels. Range 3 mg/kg to 53 %	Volatile samples may not be suitable. Standard and sample matrix must be matched for C-H ratio. Expensive instrument	See the test method	See the test method	~3–5 % relative bias
D 2784	Oxy-hydrogen burner or lamp combustion, and $BaClO_4$ titration or turbidiometry	For LPG only >1 ppm sulfur	>100 ppm halogens interfere	Not available	Not available	Not available
D 3120	Oxidative pyrolysis and microcoulometry	3–100 ppm S in light liquid hydrocarbons boiling range 26–274°C	>10*X* cl, >1000*X* N, and >500 ppm heavy metals interfere	28 %	38 %	Not available
D 3246	Oxidative pyrolisis and microcoulometry	1.5–100 ppm S in petroleum gas	>10*X* Cl, >1 % N, and >500 ppm heavy metals interfere	0.4 ppm	5 ppm	Not available
D 4045	Hydrogenolysis and rateometric colorimetry	0.02–10 ppm S in liquids with boiling points 30–371°C, for example, napthas, kerosene, alcohol, steam condensate, distillates, jet fuel, benzene, toluene		$0.16X^{0.5}$	$0.26X^{0.5}$	Not available

TABLE 11—*(Continued.)*

Test Method	Technique	Scope	Limitation	Repeatability	Reproducibility	Bias
D 4294	Energy dispersive XRF	150 ppm–5 % in Hydrocarbons such as diesel, naptha, kerosene, residuals, base oils, hydraulic oils, jet fuel, crude oils, unleaded gasoline, M–85 and M–100 fuels	Spectral interference from >0.1X of water, lead alkyls, Si, P, Ca, K, halides. Matrix effects Oxygen interferes in oxygenates	0.02894 $(X+0.1691)$	0.1215 $(X+0.0551)$	None
D 4927	Wavelength dispesive XRF	Additives, lube oils with 0.01 to 2.0 % sulfur	Spectral and matrix interferences can be compensated	See the test method	See the test methods	Not available
D 4951	ICP-AES	Lube oils and additives	Viscosity index improver gives low bias, but can be suppressed	0.016 for oils; 0.14 for additives	0.061 for oils; 0.372 for additives	
D 5185	ICP-AES	Used lube oils, base oils with 900–6000 ppm S	Same as D 4951. Particulates will give low results	$0.49X^{0\ 81}$	$1.2X^{0.75}$	None
D 5453	High temperature combustion-UV fluorescence detection	Liquid hydrocarbons boiling at 25–400°C and viscosities 0.2–10 cSt at room temperature Naptha, distillates, motor fuels, oils containing 1–8000 ppm S	>0.35 halogens interfere	$0.1867X^{0.63}$	$0.2217X^{0.92}$	None
D 6334	Wavelength dispersive XRF	15–940 ppm S in gasoline and oxygenate blends	Standards need to be matrix matched for example, oxygenates, gasohols	$0.04(X+97.29)$	$0.1182(X+54.69)$	None
D 6428	Oxidative combustion-EC detection	0.05–100 ppm S in liquid aromatic hydrocarbons	Moisture produced in combustion needs to be removed before detection	0.006 at 1 ppm S; 7.64 at 80 ppm S	Not available	Not available
D 6443	Wavelength dispersive XRF	Lube oils and additives with 0.5–1 % S	Spectral interference from Mo and Pb Other elements interference reduced by alpha corrections			None
D 6445	Energy dispersive XRF	Gasoline 48–1000 ppm S	See D 4294	$12.3(X+10)^{0.1}$	$36.26(X+10)^{0.1}$	Not available
D 6667	Combustion Ultra Violet-Fluorescence detection	Gaseous Hydrocarbons and LPG with 1–100 mg/kg sulfur		$0.1152X$	$0.3130X$	Within method repeatability
D 6920	Oxidation combusion-Electrochemical detection. Counterpart of Test Method D 6428	Naphthas, RFG distillates, diesels, biodiesels, and motor fuels. In 1–100 mg/kg range sulfur in gasoline type products, and in 1–40 mg/kg range for diesel type products	Moisture produced in combustion interferes if not removed. Equivalent amount of nitrogen may also interfere	Gasoline: $0.3395X^{0.7739}$; Diesel: 0.1960 $(X+0.6179)^{0.9022}$	Gasoline: $1.6384X^{0.7739}$; Diesel: 1.3028 $(X+0.6179)^{0.9022}$	None
D 7039	Monochromatic WDXRF	Gasoline and diesels in 2–500 mg/kg range of sulfur	Matrix matching necessary	Gasoline: $0.555X^{0.5}$; Diesel: $0.54X^{0.496}$	N/A	N/A

TABLE 11—*(Continued.)*

Test Method	Technique	Scope	Limitation	Repeatability	Reproducibility	Bias
D 7041	On-line GC Flame Photometric detector	Process feeds and finished products with final boiling point <450°C. Range 0.5–100 mg/kg sulfur		Gasoline: 0.53 Diesel: $0.207X^{0.2594}$	Gasoline: $0.0657 (X+28.626)$; Diesel: $1.9771X^{0.2594}$	N/A
D 7212	EDXRF using low background proportional counter	Fuels in the range 7 to 50 mg/kg of sulfur	Matrix matching required. Lead alkyls, Si, K, P, Ca, and halides interfere if present in >10 mg/kg level.	$1.6196X^{0.1}$	$3.7668X^{0.1}$	
D 7220	EDXRF with polarization X-ray	Fuels in the range 6 to 60 mg/kg sulfur	Matrix matching required	$1.0348X^{0.25}$	$2.0591X^{0.25}$	

SULFUR DETERMINATION BY HIGH TEMPERATURE METHOD:

D 1552

(Equivalent Test Method: AFNOR M07-025)

This test method covers three procedures applicable to petroleum products including lubricating oils containing additives and additive concentrates. This test method is applicable to samples boiling above 177°C and containing not less than 0.06 mass % sulfur. Petroleum coke containing up to 8 mass % sulfur can be analyzed.

For the iodate method, chlorine in concentrations <1 mass % does not interfere. The isoprene rubber method can tolerate somewhat higher levels. Nitrogen when present >0.1 mass % may interfere with the iodate method; the extent being dependent on the types of nitrogen compounds as well as the combustion conditions. It does not interfere in the IR method. The alkali and alkaline earth metals, zinc, potassium, and lead do not interfere with either method.

TEST SUMMARY

In the iodate detection system, the sample is burned in a stream of oxygen at a sufficiently high temperature to convert about 97 % of the sulfur to sulfur dioxide. A standardization factor is used to obtain accurate results. The combustion products are passed into an absorber containing an acidic solution of potassium iodide and starch indicator. A faint blue color is developed in the absorber solution by the addition of standard potassium iodate solution. As combustion proceeds, bleaching the blue color, more iodate is added. From the amount of standard iodate consumed during the combustion, the sulfur content of the sample is calculated.

In the IR detection system, the sample is weighed into a special ceramic boat which is then placed into a combustion furnace at 1371°C in an oxygen atmosphere. Most of the sulfur present is converted to sulfur dioxide which is then measured with an infrared detector after moisture and dust are removed by traps. The calibration factor is determined using standards approximating the material to be analyzed.

TEST PRECISION

	Repeatability		Reproducibility	
Sulfur, Mass	Iodate	IR	Iodate	IR
0.0 to 0.5	0.05	0.04	0.08	0.13
0.5 to 1.0	0.07	0.07	0.11	0.21
1.0 to 2.0	0.10	0.09	0.17	0.27
2.0 to 3.0	0.16	0.12	0.26	0.38
3.0 to 4.0	0.22	0.13	0.40	0.44
4.0 to 5.0	0.24	0.16	0.54	0.49

For petroleum coke, the repeatability was 0.05 X and reproducibility 0.22 X where X is the average of two test results.

The bias of this procedure has not been determined.

SULFUR DETERMINATION BY HYDROGENOLYSIS AND RATEOMETRIC COLORIMETRY:

D 4045

This test method is valid in the range 0.02 to 10.00 mg/kg of sulfur in petroleum products. It may be extended to higher concentrations by dilution. This test method is applicable to liquids with boiling points between 30 to 371°C. These include naptha, kerosene, alcohol, steam condensate, various distillates, jet fuel, benzene, and toluene.

TEST SUMMARY

This test method is based on an instrument available from Houston Atlas, Inc. The sample is injected at a constant rate into a flowing hydrogen stream in a hydrogenolysis apparatus. The sample and hydrogen are pyrolyzed at 1300°C or higher, to convert sulfur compounds to hydrogen sulfide. Readout is by the rateometric detection of the colorimetric reaction of hydrogen sulfide with lead acetate. Condensable compounds are converted to gaseous products such as methane during the hydrogenolysis.

TEST PRECISION

Repeatability:	$0.16X^{0.5}$
Reproducibility:	$0.26X^{0.5}$

Bias of this test method has not been determined.

SULFUR DETERMINATION SULFUR BY LAMP METHOD:

D 1266

(Equivalent Test Methods: IP 107 and AFNOR M07-031)

This test method is valid in the concentration range of 0.01 to 0.04 mass % of sulfur in liquid petroleum products. By using a special sulfate analysis procedure the determination can be extended to as low as 5 mg/kg of sulfur. The direct burning procedure is applicable to gasoline, kerosene, naptha, and other liquids that can be burned completely in a wick lamp. The blending procedure is applicable to gas oils, distillate fuel oils, napthenic acids, alkyl phenols, high sulfur content petroleum products, and other materials that cannot be burned satisfactorily by the direct burning procedure.

Phosphorus compounds normally present in commercial gasoline do not interfere. A correction is needed for the small amount of acid resulting from the combustion of lead antiknock fluids in gasolines. Appreciable concentrations of acid- or base-forming elements from other sources interfere when the titration procedure is employed, since no correction is provided in these cases.

TEST SUMMARY

A sample is burned in a closed system using a suitable lamp and an artificial atmosphere composed of 70 % carbon dioxide and 30 % oxygen to prevent formation of nitrogen oxides. The oxides of sulfur produced are absorbed and oxidized to sulfuric acid by means of hydrogen peroxide solution which is then flushed with air to remove dissolved carbon dioxide. Sulfur as sulfate in the absorbent is determined acidimetrically by titration with standard NaOH solution, or gravimetrically by precipitation as barium sulfate.

Alternatively, the sample may be burned in air and the sulfur as sulfate in the absorbent be determined by precipitating as barium sulfate for weighing. For sulfur content below 0.01 mass %, it is necessary to determine sulfur in the absorber solution turbidimetrically as barium sulfate.

TEST PRECISION

Test Method	Repeatability	Reproducibility
D 1266: 0.01 to 0.04 m % Sulfur	0.005	$0.10+0.25\,X$
D 1266, Annexe Al: Sulfur 5 to 80 ppm	$0.116\,X$	$0.145\,X$
Sulfur over 80 to 280 ppm	$(0.01\,X)+8.5$	$(0.508\,X)-45.4$

Where X is the mean sulfur concentration in mass % for Test Method D 1266 and in mg/kg for Test Method D 1266, Annexe Al.

The bias of these test methods has not been determined.

SULFUR DETERMINATION IN LIQUID PETROLEUM GASES (LPG):

D 2784

This test method is valid for sulfur levels of >1 μg/g of sulfur in liquefied petroleum gases (LPG). The samples should not contain more than 100 μg/g of chlorine. Stringent techniques must be employed and all possible sources of sulfur contamination must be eliminated to achieve the quantitative detectibility that this test method is capable of. In particular, cleaning agents, such as common household detergents, which contain sulfates, should be avoided.

TEST SUMMARY

The sample is burned in an oxy-hydrogen burner or in a lamp in a closed system in a carbon dioxide oxygen atmosphere. The latter is not recommended for trace quantities of sulfur due to inordinately long combustion times needed. The sulfur oxides produced are absorbed and oxidized to sulfuric acid in a hydrogen peroxide solution. The sulfate ions are then determined by either titrating with barium perchlorate solution using a thorinmethylene blue mixed indicator, or by precipitating as barium sulfate and measuring the turbidity of the precipitate with a photometer.

TEST PRECISION

No data on either precision or bias of this test method are available.

SULFUR IN GASOLINE AND DIESEL FUEL BY MONOCHROMATIC WDXRF:

D 7039

EXPLANATION

This test method determines total sulfur in gasolines and diesels with minimal sample preparation and analyst involvement. A typical time for each analysis is 2 to 3 min. The scope of the method ranges from 2 to 500 mg/kg sulfur. The pooled limit of quantitation is estimated to be 4 mg/kg. Gasoline samples containing oxygenates may be analyzed with this test method provided the matrix of the calibration standards is either matched to the sample matrices or a matrix correction is applied to the results. Gasolines and diesels containing sulfur concentration above 50 mg/kg can be analyzed after dilution with appropriate solvent.

TEST SUMMARY

A monochromatic X-ray beam with a wavelength suitable to excite the K-shell electrons of sulfur is focused onto a test specimen contained in a sample cell. The fluorescent K alpha radiation at 0.5373 nm emitted by sulfur is collected by a fixed monochromator analyzer. The intensity (counts per second) of the sulfur X-rays is measured using a suitable detector and converted to the concentration of sulfur (mg/kg) in a test specimen using a calibration equation. Excitation by monochromatic X-rays reduces background, simplifies matrix correction, and increases the signal to background ratio compared to polychromatic excitation used in conventional WDXRF techniques.

INTERFERENCES—Differences between the elemental composition of the test samples and the calibration standards can result in biased sulfur determinations. For diesels and gasolines, the only important element contributing to bias resulting from differences in the matrices of the calibrants and test samples are hydrogen, carbon, and oxygen. A matrix correction factor can be used to correct this bias. For practical purposes, the matrices of the test samples and the calibrants are considered as matched when the calculated correction factor is within 0.98 to 1.04. No matrix correction is required within this range. For most testing, matrix correction can be avoided with a proper choice of calibrants.

To minimize any bias in the results, calibration standards prepared from sulfur-free base materials of the same or similar elemental composition as the test samples should be used. When diluting samples, a diluent with an elemental composition the same or similar to the base material used for preparing the calibration standards should be used. Fuels containing oxygenates may be analyzed using calibration standards containing the same amount of the same oxygenate in the test fuel.

TEST PRECISION

Gasoline Repeatability:	$0.555 \times X^{0.5}$
Diesel Repeatability:	$0.54 \times X^{0.496}$

Test method reproducibility data or bias are not yet available.

SULFUR IN PETROLEUM PRODUCTS BY ON-LINE GC WITH FPD:
D 7041

EXPLANATION

Low levels of sulfur in process feed stocks can poison expensive catalysts used in petroleum refining processes. This test method can be used to determine total sulfur levels in these process feeds and finished products that fall within the scope of this method. This test method covers the liquid hydrocarbon samples with a final boiling point less than 450°C (such as motor fuels and oils) by gas chromatography using a flame photometric detector. This test method is applicable to total sulfur levels from 0.5 to 100 mg/kg of sulfur. The pooled limit of detection is determined to be 1 mg/kg. Samples can also be tested at other higher levels.

TEST SUMMARY

A fixed amount of sample is injected into the gas chromatograph where it is vaporized. The air carrier stream carries the vaporized sample into a high temperature zone (>900°C) where the compounds present in the sample are oxidized. Sulfur compounds are converted to SO_2. The carrier stream carries the oxidation components onto a chromatographic column where they are separated, and the SO_2 is quantified by the flame photometric detector. Calibration of the detector is achieved by the use of an appropriate external standard.

TEST PRECISION

Product	Repeatability	Reproducibility
Gasoline	0.53	$0.0657(X+28.626)$
Diesel	$0.2070\ X^{0.2594}$	$1.9771\ X^{0.2594}$

Where X is the mean of results. All values are in mg/kg of sulfur.

SULFUR DETERMINATION BY OXIDATIVE COMBUSTION AND ELECTROCHEMICAL DETECTION:
D 6428

This test method is valid for sulfur levels of 0.05 to 100 mg/kg in liquid aromatic hydrocarbons, their derivatives, and related chemicals. Virtually all sulfur compounds will be detected by this technique.

TEST SUMMARY

The sample is injected at a controlled rate into a stream of inert gas (helium or argon) or inert gas mixed with oxygen. The sample is vaporized and carried into a high temperature zone (>900°C) where oxygen is introduced. Sulfur compounds are converted to sulfur dioxide which is then reacted with the sensing electrode in a 3-electrode electrochemical cell. This reaction produces a measurable current that is directly proportional to the amount of sulfur in the original sample.

TEST PRECISION

Based on very limited data, the following precision estimates are reported:

Sulfur Level, mg/kg	Standard Deviation, mg/kg
1.01	0.02
80.0	2.76

SULFUR IN PETROLEUM PRODUCTS BY OXIDATIVE COMBUSTION WITH ELECTROCHEMICAL DETECTION: D 6920

EXPLANATION

This test method covers the determination of total sulfur in liquid hydrocarbons (e.g., naphthas, distillates, reformulated gasolines, diesels, biodiesels, and motor fuels) boiling in the range from approximately 25–400°C, with viscosities between approximately 0.2 and 10 cSt at room temperature, and containing approximately 1–100 mg/kg of total sulfur in gasoline type products, and approximately 1–40 mg/kg in diesel type products. The detector response for this technique within the scope of this test method is linear with sulfur concentration. The pooled limit of quantitation of this method is 3 mg/kg for gasoline and <0.5 mg/kg for diesel samples. This test method is equivalent to Test Method D 6428 which was at one time designated by U.S. EPA as the preferred method for determination of ultra low sulfur in diesels.

TEST SUMMARY

The sample is injected at a controlled rate into a stream of inert gas (helium or argon) or inert gas mixed with oxygen. The sample is vaporized and carried into a high temperature zone (>900°C) where oxygen is introduced. Sulfur compounds, present in the specimen are converted to SO_2. This is reacted with the sensing electrode in a 3-electrode electrochemical cell. This reaction produces a measurable current that is directly proportional to the amount of sulfur in the original sample. The reactions that occur are as follows:

Combustion: $R-S+O_2@>900°C \rightarrow CO_2+H_2O+SO_2 + \text{OXIDES}$

Detection: $SO_2+2H_2O \rightarrow H_2SO_4+2H^++2e^-$

INTERFERENCES—Moisture produced during the combustion step can interfere if not removed prior to the detector. Equivalent amounts of nitrogen in the sample may also interfere.

TEST PRECISION

Sample	Sulfur Range	Repeatability	Reproducibility
Gasolines	3–100 mg/kg	$0.3395\ X^{0.7739}$	$1.6384\ X^{0.7739}$
Diesels	1–40 mg/kg	$0.1960\ (X+0.61789)^{0.9022}$	$1.3028\ (X+0.61789)^{0.9022}$

Where X is the average of two sulfur results in mg/kg. It was found that the two instruments used in the interlaboratory study—APS and Antek—gave significantly different precisions.

Based on the analyses of NIST Standard Reference Materials, there was no significant bias for the test method on either instrument for diesel SRM 2723a and gasoline SRM 2299. However, for gasoline SRM 2298, there was –0.78 mg/kg bias for this test method on both instruments.

SULFUR IN FUELS BY EDXRF USING A LOW BACKGROUND PROPORTIONAL COUNTER: D 7212

EXPLANATION

This test method measures total sulfur in the concentration range 7 to 50 mg/kg automotive fuels with a typical analysis time around 10 min per sample. This test method uses an energy dispersive X-ray fluorescence spectrometer (EDXRF). The pooled limit of quantitation of the method is 7 mg/kg.

TEST SUMMARY

The sample is placed in the beam emitted from an X-ray source with titanium target and primary filtration so that excitation is by essentially monochromatic radiation of 4.51 keV. A low background proportional counter measures the intensity of the fluorescent sulfur K series intensity and argon

K series intensity (from residual air) and the accumulated counts are compared with counts from previously prepared calibration standards to obtain the sulfur concentration in mg/kg. If chlorine is expected to be present in some samples then other regions of the spectrum must be measured to provide compensation for spectral overlap.

INTERFERENCES—Spectral interferences result when sample component element or elements emit X-rays that the detector cannot resolve from sulfur X-ray emission. This results in overlapping of peak lines. Such effects may result from the presence of lead alkyls, silicon, phosphorus, calcium, potassium, and halides in the sample if their aggregate concentration is more than 10 mg/kg. The most likely interference is chlorine that has been found in biodiesel derived from recycled waster vegetable oil. The presence of oxygenates or water may alter the sensitivity for sulfur. Follow the manufacturers' operating guide to compensate for these interferences.

TEST PRECISION

Repeatability:	1.6196 $X^{0.1}$
Reproducibility:	3.7668 $X^{0.1}$

Where X is the average of two results.

Based on the analysis of a diesel fuel certified standard no significant bias was found for this method.

SULFUR IN FUELS BY POLARIZATION XRF: D 7220

EXPLANATION

This test method uses an energy dispersive X-ray fluorescence (EDXRF) technique for the determination of total sulfur in automotive fuels with a concentration range of 6 to 50 mg/kg. The pooled limit of quantitation is 6 mg/kg sulfur. A typical analysis time is 200 to 300 sec per sample.

TEST SUMMARY

The sample is placed in the polarized X-ray beam, and the peak area of the sulfur k line at 2.307 keV is measured. The background spectrum, measured with a sulfur free white oil or other matrix matching blank sample is adapted to the measurement spectrum using adjustment regions following the instrument manufacturers' instructions, and then subtracted from the measured spectrum. The resultant net counting rate is then compared to a previously prepared calibration curve or equation to obtain the concentration of sulfur in mg/kg.

INTERFERENCES—The sample and standard matrix must be well matched. Matrix mismatch can be caused by C/H ratio differences between samples and standards or by the presence of other heteroatoms.

TEST PRECISION

Repeatability:	1.0348 $X^{0.25}$
Reproducibility:	2.0591 $X^{0.25}$

Where X is the average of two results in mg/kg.

Based on the analysis of a certified fuel reference sample, the method showed no apparent bias.

SULFUR DETERMINATION BY OXIDATIVE MICROCOULOMETRY: D 3120

This test method is valid at sulfur levels of 3.0 to 100 ppm in light liquid hydrocarbons boiling in the range from 26 to 274°C. Samples with higher sulfur levels may be analyzed after proper dilutions. This test method is applicable in the presence of total halides up to 10 times the sulfur concentration, and total nitrogen concentration up to 1000 times the sulfur level. The method is not applicable in the presence of total heavy metal concentrations (for example, nickel, vanadium, lead, etc.) in excess of 500 ppm . Stringent techniques must be employed and all possible sources of sulfur contamination must be eliminated to achieve the quantitative detectability that this test method is capable of.

TEST SUMMARY

A liquid sample is injected into a combustion tube maintained at about 800°C in a flowing stream of gas mixture of 80 % oxygen and 20 % inert gas such as nitrogen or argon, etc.

Oxidative pyrolysis converts the sulfur to sulfur dioxide, which then flows into a tiration cell where it reacts with triiodide ion present in the electrolyte. The triiodide thus consumed is coulometrically replaced and the total current required to replace it is a measure of the sulfur present in the injected sample.

TEST PRECISION

	Repeatability	Reproducibility
Case I	$0.2802\ X^{0.7901}$	$0.5793\ X^{0.7901}$
Case II-Gasoline	$0.03194\ (X+7.4502)^{1.3226}$	$0.1470\ (X+7.4502)^{1.3226}$
-Diesel	$0.08520\ (X+0.65758)$	$0.5152\ (X+0.65758)$

Where X is the average of duplicate results in mg/kg

A pooled limit of quantitation (PLOQ) of ~3 mg/kg for gasoline samples and ~5 mg/kg for diesel sample types was obtained.

SULFUR DETERMINATION BY OXIDATIVE MICROCOULOMETRY:

D 3246

(Equivalent Test Methods: IP 373 and AFNOR M07-052)

This test method is valid in the sulfur concentration range of 1.5 to 100 ppm in hydrocarbon products that are gaseous at normal room temperature and pressure. This test method is applicable in the presence of total halide concentration of up to 10 times the sulfur level and total nitrogen content of up to 1.0 %. Free nitrogen does not interfere. This test method is not applicable in the presence of total heavy metal concentration (for example, nickel, vandium, iron, lead, etc.) in excess of 500 ppm. Stringent techniques should be employed and all possible sources of sulfur contamination must be eliminated to achieve the quantitative detectability that this test method is capable of.

TEST SUMMARY

See details under Test Method D 3120.

TEST PRECISION

In the 0 to 10 mg/kg level of sulfur, the test repeatability is 0.4 and reproducibility is 5 mg/kg.

The bias of this test method has not been determined.

SULFUR DETERMINATION BY GC-SULFUR DETECTOR:

D 5623

Frequently the knowledge of individual sulfur compounds present in a product is more useful than the total amount of sulfur present. This test method covers the determination of volatile sulfur compounds in light petroleum liquids. This test method is applicable to distillates, gasoline motor fuels, including those containing oxygenates, and other petroleum liquids with the FBP of approximately 230°C or lower at atmospheric pressure. Generally, this test method can determine individual sulfur species at levels of 0.1 to 100 mg/kg. This test method does not purport to identify all individual sulfur compounds present. Also, some sulfur compounds, such as hydrogen sulfide and mercaptans, are reactive and their concentration in samples may change during sampling and analysis. Total sulfur content of the sample can be estimated from the sum of the individual compounds determined. However, this test method is not the preferred method for the determination of total sulfur in a petroleum liquid.

TEST SUMMARY

The sample is analyzed by gas chromatography with an appropriate sulfur selective detector. Calibration is done by an appropriate internal or external standard. All sulfur compounds are assumed to produce equivalent response as sulfur. As sulfur compounds elute from the gas chromatographic column, they are quantified by a sulfur selective chemiluminescence detector that produces a linear and equimolar response to sulfur compounds.

TEST PRECISION

Standardization	Compound	Range, mg/kg	Repeatability, mg/kg	Reproducibility, mg/kg
Internal	Single Component	1 to 100	0.11 X	0.42 X
	Total Sulfur	10 to 200	0.12 X	0.33 X
External	Single Component	1 to 100	0.31 X	0.53 X
	Total Sulfur	10 to 200	0.24 X	0.52 X

Bias of this test method has not been determined.

SULFUR DETERMINATION BY ULTRAVIOLET FLUORESCENCE METHOD:
D 5453

EXPLANATION

Some process catalysts used in petroleum and chemical refining may be poisoned when trace amounts of sulfur bearing materials are contained in the feedstocks. This test method can be used to determine sulfur in process feeds and may also be used to control sulfur in finished products.

This test method covers the determination of total sulfur in liquid hydrocarbons, boiling in the range from approximately 25°C to 400°C, with viscosities between approximately 0.2 and 10 cSt (mm^2/S) at room temperature. This test method is applicable to naphthas, distillates, motor fuels oils, engine oils, ethanol, FAME, and engine fuels such as gasoline, RFG, diesel, biodiesel, and jet fuel containing 1.0 to 8000 mg/kg total sulfur. The pooled limit of quantitation ranges from <1 to <5 mg/kg depending on the matrix being analyzed.

This test method is applicable for total sulfur determination in liquid hydrocarbons containing less than 0.35 mass % halogen(s).

The pooled limit of quantitation ranges from <1 to <5 ppm depending on the matrix analyzed.

TEST SUMMARY

A hydrocarbon sample is directly injected or placed in a sample boat. The sample or boat, or both, enters into a high temperature combustion tube where the sulfur is oxidized to sulfur dioxide (SO_2) in an oxygen rich atmosphere. Water produced during the sample combustion is removed and the sample is next exposed to ultraviolet (UV) light. The SO_2 absorbs the energy from the UV light and is converted to excited sulfur dioxide (SO_2). The fluorescence emitted from the excited SO_2 as it returns to a stable state is detected by a photomultiplier tube and the resulting signal is a measure of the sulfur contained in the sample.

TEST PRECISION

	Repeatability	Reproducibility
Less than 400 mg/kg	$0.1788\ X^{0.75}$	$0.5797\ X^{0.75}$
Greater than 400 mg/kg	0.02902 X	0.1267 X
Gasoline	$0.04356(X+14.844)$	$0.3170(X+6.8847)$
Diesel	$0.02777(X+28.158)$	$0.3659(X+2.1101)$

Where X is the mean result in mg/kg.

This test method has no bias based on the analysis of standard reference materials.

TOTAL VOLATILE SULFUR IN GASEOUS HYDROCARBONS AND LPG USING COMBUSTION UV FLUORESCENCE DETECTION:

D 6667

EXPLANATION

The sulfur content of LPG, used for fuel purposes, contributes to SO_x emissions and can lead to corrosion in engine and exhaust systems. Some process catalysts used in petroleum and chemical refining can be poisoned by sulfur bearing materials in the feed stocks. This test method can be used to measure sulfur in process feeds, finished products, and can also be used for compliance determinations when acceptable to a regulatory agency. This test method is applicable to analysis of natural, processed, and final product materials (gaseous hydrocarbons and LPG) containing sulfur in the range 1 to 100 mg/kg. This test method may not detect sulfur compounds that do not vaporize under the test conditions. This test method is applicable for total volatile sulfur determination in LPG containing less than 0.35 m/m % halogen(s).

TEST SUMMARY

A heated sample valve is used to inject gaseous samples. LPG samples are injected by a sample valve connected to a heated expansion chamber. The gaseous sample then enters a high temperature combustion tube where sulfur is oxidized to SO_2 in an oxygen rich atmosphere. Water produced during the sample combustion is removed, and the sample combustion gases are next exposed to ultraviolet light. The SO_2 absorbs the energy from the UV light and is converted to an excited SO_2^*. Fluorescence emitted from the excited SO_2^* as it returns to a stable state SO_2 is detected by a photomultiplier tube. The resulting signal is a measure of the sulfur contained in the sample.

TEST PRECISION

Repeatability:	0.1152 (X)
Reproducibility:	0.3130 (X)

Where X is the average of two test results.

The bias of this test method was within the repeatability of this test method.

SULFUR DETERMINATION SULFUR BY WAVELENGTH DISPERSIVE X-RAY FLUORESCENCE (WD-XRF):

D 2622

(Equivalent Test Methods: DIN 51400 T6 and JIS K 2541)

This test method is applicable for the analysis of diesel fuel, jet fuel, kerosene, other distillate oil, naptha, residual oil, lubricating base oil, hydraulic oil, crude oil, unleaded gasoline, M-85, and M-100. This test method has been tested at least in the sulfur concentration range of 3 mg/kg to 5.3 mass %. Samples with higher sulfur levels can be analyzed with proper dilution. Volatile materials, such as high vapor pressure gasolines or light hydrocarbons, may not meet the stated precision limits of this test method because of the selective loss of light materials during the analysis. When the elemental composition (excluding sulfur) of samples differs significantly from the standards, errors in the analysis can occur. To maintain the same carbon-to-hydrogen ratio, the standard and the sample matrix must be well matched for this analysis. M-85 and M-100 fuels contain 85 and 100 % methanol, respectively. Their high oxygen content leads to significant absorption of sulfur Kα radiation. To compensate for this, either correction factors must be applied or calibration standards matching the samples need to be used. Compared to other sulfur test methods, this method has high throughput, minimal sample preparation, and excellent precision. It is capable of determining sulfur over a wide range of concentrations. The equipment required, however, is significantly more expensive than for other methods.

TEST SUMMARY

A sample is placed in an X-ray beam, and the peak intensity of the sulfur Kα line at 5.373 Å is measured. The background intensity, measured at 5.190 Å is subtracted from the peak intensity. The resultant net counting rate is then compared to a previously prepared calibration curve or equation to obtain the sulfur concentration in mass percent.

TEST PRECISION

Products	Range, m%	Repeatability	Reproducibility
Case I	0.006–5.3	$0.02651\ X^{0.9}$	$0.0913\ X^{0.9}$
Case II	0.0003–0.093	$0.00736(X+0.0002)^{0.4}$	$0.0105(X+0.0002)^{0.4}$
Case III	0.0024–0.0080	$0.02438(X+0.012469)$	$0.04795(X+0.012469)$

Where X is the mean sulfur concentration in mass %.

Case I includes distillates, kerosenes, residual oils, and crude oils.

Case II includes gasolines.

Gasohols were not included.

Case III included low level sulfur gasoline and diesel sample.

Based on the analysis of NIST SRM materials, there was significant bias in the analysis, but this was eliminated after correction for C/H ratio.

SULFUR DETERMINATION BY ENERGY DISPERSIVE X-RAY FLUORESCENCE (ED-XRF):

D 4294

(Equivalent Test Methods: IP 336, ISO 8754, and AFNOR M07-053)

This test method is applicable to all the petroleum products mentioned in Test Method D 2622 (see previously). The applicable concentration range of sulfur is 0.015 to 5.0 mass %. There can be interferences from spectral lines and matrix matching. Generally, spectral interference will arise from elements at concentrations greater than one-tenth of the sulfur concentration (for example, water, lead, alkyls, silicon, potassium, calcium, phosphorous, and halides). Matrix interferences arise from different C/H ratio or oxygen present in the standard and samples. Both types of interferences are compensated for in the modern instruments with the use of built-in software. High oxygen-containing fuels M-85 and M-100 should be analyzed using calibration standards that match the sample matrix. Suspended water if any in the sample must be removed or thoroughly homogenized and immediately analyzed. Compared to other test methods for sulfur determination, this method has high throughput (2 to 4 min per sample), minimal sample preparation, good precision, and is capable of determining sulfur over a wide concentration range. The equipment specified in most cases is less costly than that required for alternative methods.

TEST SUMMARY

The sample is placed in a beam emitted from an X-ray source. The resultant excited characteristic X radiation is measured, and the accumulated count is compared with counts from a previously prepared calibration standard to obtain the sulfur concentration. Two groups of calibration standards are required to span the concentration range, one from 0.015 to 0.1 %, and the other from 0.1 to 5.0 %.

TEST PRECISION

Repeatability:	$0.02894(X+0.1691)$
Reproducibility:	$0.1215(X+0.05555)$

Where X is the average of two results.

There is no bias.

SULFUR DETERMINATION IN GASOLINE BY ENERGY-DISPERSIVE X-RAY FLUORESCENCE SPECTROMETRY: D 6445

EXPLANATION

The quality of gasoline is related to the amount of sulfur present. Knowledge of sulfur content is necessary for processing purposes. Regulations by federal, state, and local agencies restrict the amount of sulfur present in gasoline as it affects performance characteristics and potential corrosion problems and emission levels. Certain jurisdictions may restrict the amount of sulfur in gasoline to prevent or limit environmental pollution caused by sulfur oxide emissions formed from sulfur in the fuel during combustion of fuel.

This is a rapid and precise method for measurement of sulfur in petroleum products with a minimum of sample preparation. This method covers the sulfur measurement in the range 49 to 1000 mg/kg in nonleaded gasoline and gasoline-oxygenate blends.

TEST SUMMARY

The sample is placed in a beam emitted from an X-ray source. The resultant excited characteristic X radiation is measured, and the accumulated count is compared with counts from previously prepared calibration standards to obtain the sulfur concentration.

Compounds such as oxygenates present in the sample can affect the apparent sulfur readings. Such matrix effects can be compensated by many modern instruments.

TEST PRECISION

Repeatability:	$12.30(X+10)^{0.1}$
Reproducibility:	$36.26(X+10)^{0.1}$

Where X is the sulfur concentration in mg/kg.

Bias is not known.

SULFUR DETERMINATION IN GASOLINE BY WD-XRF: D 6334

This test method is applicable to gasoline and gasoline-oxygenate blends in the sulfur concentration range of 15 to 940 mg/kg. Similar to other XRF methods just described, the standards and samples must match in their matrix composition.

TEST SUMMARY

The sample is placed in the X-ray beam, and the intensity of the sulfur Kα line at 5.373 Å is measured. The intensity of the corrected background, measured at the wavelength of 5.190 Å is subtracted from this intensity. The resultant net counting rate is then compared to a previously prepared calibration curve or equation to obtain the concentration of sulfur in mg/kg.

TEST PRECISION

In the 15 to 940 mg/kg range, this test method has a repeatability and a reproducibility $0.0400\times(X+97.29)$ and $0.1182(X+54.69)$, respectively, where X is the mean sulfur concentration in mg/kg.

There was no bias in the results between this test method and other Test Methods D 2622, D 3210, D 4045, D 4294, and D 5453.

OXIDATION STABILITY OF LUBRICANTS BY TFOUT CATALYST B:
D 7098

EXPLANATION

This test method was originally developed to evaluate oxidation stability of lubricating base oils combined with additives chemistries similar to those found in gasoline engine oils and services. This test method is useful for screening formulated oils before engine testing. Within similar additive chemistries and base oil types, the ranking of oils in this test appears to be predictive of ranking in certain engine tests. When oils having different additive chemistries or base oil types are compared, results may or may not reflect results in engine tests. This test method covers the oxidation stability of lubricants by thin film oxygen uptake (TFOUT) catalyst B.

TEST SUMMARY

The test oil is mixed in a glass container with four other liquids used to simulate engine conditions: an oxidized/nitrated fuel component, a mixture of soluble metal naphthenates (lead, iron, manganese and tin naphthenates, a nitro-paraffinic compound, and Type II reagent water. The glass container with the oil mixture is placed in a pressure vessel equipped with a pressure sensor. The pressure vessel is sealed, charged with oxygen to a pressure of 620 kPa and placed in an oil bath at 160°C at an angle of 30° from the horizontal. The pressure vessel is rotated axially at a speed of 100 r/min forming a thin film of oil within the glass container resulting in a relatively large oil-oxygen contact area.

The pressure of the pressure vessel is recorded continuously from the beginning of the test, and the test is terminated when a rapid decrease of the pressure vessel is observed. The period of time that elapses between the time when the pressure vessel is placed in the oil bath and the time at which the pressure begins to decrease is called the oxidation induction time and is used as a measure of the relative oil oxidation stability.

TEST PRECISION

Repeatability:	14 % of mean
Reproducibility:	39 % of mean
Bias	Cannot be determined

SURFACE WAX COATING ON CORRUGATED BOARD:
D 3521

EXPLANATION

Wax coatings are applied to corrugated board to provide a better barrier against moisture or other agents or to provide improved appearance or abrasion resistance. These performance features are influenced by the amount of wax present on the surface. During most coating operations, the major portion of the wax applied will congeal on the surface, while a minor proportion will migrate into and become embedded in the fibers of the facing. This method measures only the portion on the surface. This method is applicable to board to which wax has been applied by curtain coating, roll coating, or other methods; the substrate board may or may not contain impregnating (saturating) wax within its structure. If it is known that the specimen has coating wax only, with no internal saturating wax, the total coating wax applied may be determined by Test Method D 3344.

TEST SUMMARY

The amount of wax present as a coating on the surface is determined by delaminating the coated facing to obtain a ripple-free sheet, then scrapping off the wax using a razor blade, and calculating the wax removed.

TEST PRECISION

Repeatability:	15 % of the mean
Reproducibility:	24 % of the mean

Due to the high degree of normal variability from specimen to specimen, close precision may be difficult to obtain. In cases where close precision is needed or precision is suspect, multiple sampling and testing should be done.

This procedure has no bias because the value of the surface wax can be defined only in terms of this test method.

SURFACE WAX ON WAXED PAPER OR PAPERBOARD: D 2423

EXPLANATION

Many of the fundamental properties of waxed paper and paperboard are related to the amount of wax present as a surface film. Test methods which determine wax load by solvent extraction do not differentiate between the wax present as the surface wax layer and that which has penetrated into the substrate. This test method, which mechanically removes the wax, measures the amount on each surface of the substrate.

TEST SUMMARY

The quantity of wax present as a surface film on paper or paperboard is determined through the difference in weight of specimens before and after scraping with a razor blade.

TEST PRECISION

Repeatability:	0.976 g/m^2(0.6 lb/ream)
Reproducibility:	1.46 g/m^2(0.9 lb/ream)

This procedure has no bias because the surface wax on waxed paper or paperboard can be defined only in terms of this test method.

THERMAL CONDUCTIVITY OF LIQUIDS: D 2717

EXPLANATION

The thermal conductivity of a substance is a measure of the ability of that substance to transfer energy as heat in the absence of mass transport phenomena. It is used in engineering calculations that relate to the manner in which a given system can react to thermal stress. This test method is applicable to liquids that are:

(1) chemically compatabile with borosilicate glass and platinum,
(2) moderately transparent or absorbent to infrared radiation, and
(3) have a vapor pressure of less than 200 torr at the temperature of the test.

Materials that have vapor pressures up to 345 kPa absolute can be tested.

TEST SUMMARY

A thermal conductivity cell consisting of a straight, four-lead, platinum resistance thermometer element located concentrically in a long, small diameter, precision bore borosilicate glass tube is calibrated by accurate measurement of the cell dimensions and by determination of the temperature resistant properties of the platinum element. Thermal conductivity is determined by measurement of the temperature gradient produced across the liquid sample by a known amount of energy introduced into the cell by electrically heating the platinum element.

TEST PRECISION

Precision data are not yet available. However, preliminary estimate appears to be 10 % repeatability for the mean of the two results.

THERMAL STABILITY OF AVIATION TURBINE FUELS BY JFTOT PROCEDURE:

D 3241

(Equivalent Test Methods: IP 323, ISO 6249, JIS K 2276B, and AFNOR M07-051)

EXPLANATION

This test method covers the procedure for rating the tendencies of gas turbine fuels to deposit decomposition products within the fuel system. The test results can be used to assess the level of deposits that form when the liquid fuel contacts a heated surface at a specified temperature.

TEST SUMMARY

This test method uses the Jet Fuel Thermal Oxidation Tester (JFTOT) that subjects the fuel to conditions that can be related to those occurring in gas turbine engine fuel systems. The fuel is pumped at a fixed rate through a heater after which it enters a precision stainless steel filter where fuel degradation products may become trapped. The essential data derived are the amount of deposits on an aluminum heater tube, and the rate of plugging of a 17 μ nominal porosity precision filter.

This instrument is supplied by ALCOR Petroleum Instruments, Inc.

TEST PRECISION

The precision of this test method is currently being determined.

This test method has no bias.

THERMAL STABILITY OF AVIATION TURBINE FUELS BY HIRETS METHOD:

D 6811

EXPLANATION

The thermal stress experienced by aviation fuel in modern jet engines may lead to the formation of undesirable and possibly harmful insoluble materials, such as lacquers, on heat exchangers and control surfaces, that reduce efficiency and require extra maintenance. Aircraft fuel systems operate mainly under turbulent flow conditions. Most large scale realistic test rigs operate in the turbulent flow regime but fuel volumes are very large and test times are very long. This test method tests fuel under turbulent flow (high Reynolds number) conditions, and it gives a quantitative result under standard operating conditions of 65 or 125 min. Continuous analysis of the results during the test allows performance of the fuel to be monitored in real time thus enabling the test time to be reduced manually or automatically, if required. The results of this test method are not expected to correlate with existing test methods for all fuels, since the test methods and the operating conditions are different.

This test method covers a laboratory thermal process using a specified apparatus for measuring the tendencies of aviation turbine fuels to deposit insoluble materials and decomposition products, such as lacquers, within a fuel system.

TEST SUMMARY

Fuel is pumped at pressure, through an electrically heated capillary tube at a constant rate. The heating of the capillary tube is controlled to maintain a constant fuel temperature of $290 \pm 3°C$ at the exit of the capillary tube. A flow rate greater than 20 mL/min and the specified capillary bore of less than 300 mm ensures that turbulent flow is maintained within the capillary. The formation of lacquers and fuel degradation products act as a thermal insulator between the cooler fuel and hotter capillary tube, resulting in an increase in temperature of the capillary tube which is measured at a number of positions by a contactless pyrometer. The HiReTS Total (T) number is displayed during and at the end of the test. The HiReTS Peak (P) number can be determined from analysis of the results.

CORRELATIONS—

(1) Some test programs have shown a strong correlation between carbon burn off measurements from used capillary tubes and their respective HiReTS results.

(2) Correlation between JFTOT D 3241 and this test method is unlikely due to the difference between the two test methods, and with HiReTS operating in the turbulent flow regime using stainless tubes, and the

JFTOT operating in the laminar flow regime using aluminum tubes. A HiReTS Reynolds number of greater than 5000 can be contrasted with a JFTOT Reynolds number of 11.

TEST PRECISION

Parameter	Repeatability	Reproducibility
P65 (Peak number with a 65 min test)	$2.006\ X^{0.667}$	$3.077\ X^{0.667}$
P125 (Peak number with a 125 min test)	$2.249\ X^{0.667}$	$2.896\ X^{0.667}$
T65 (Total number with a 65 min test)	$3.123\ X^{0.72}$	$5.008\ X^{0.72}$
T125 (Total number with a 125 min test)	$1.322\ X^{0.9}$	$1.667\ X^{0.9}$

Where X is the average of results being compared. The precision applies only to HiReTS Peak numbers up to 200 and Total numbers up to 1900; higher values can be measured but the precision estimates may not apply. This test method has no bias because the result of the test is defined only in terms of this test method.

THERMAL STABILITY OF ORGANIC HEAT TRANSFER FLUIDS:

D 6743

EXPLANATION

Heat transfer fluids degrade when exposed to sufficient high temperatures. The amount of degradation increases as the temperature increases or the length of exposure increases, or both. Due to reaction and rearrangement, degradation products can be formed. Degradation products include high and low boiling components, gaseous decomposition products, and products that cannot be evaporated. The type and content of degradation products produced will change the performance characteristics of a heat transfer fluid. In order to evaluate thermal stability, it is necessary to quantitatively determine the mass percentages of high and low boiling components, as well as gaseous decomposition products and those that cannot be vaporized, in the thermally stressed heat transfer fluid. This test method differentiates the relative stability of organic heat transfer fluids at elevated temperatures in the absence of oxygen and water under the conditions of the test.

The users should determine to their own satisfaction whether the results of this test method correlate to field performance. Heat transfer fluids in industrial plants are exposed to a variety of additional influencing variables. Interaction with the plant's materials, impurities, heat build-up during impaired flow conditions, the temperature distribution in the heat transfer fluid circuit, and other factors can also lead to changes in the heat transfer fluid. This test method provides an indication of the relative thermal stability of a heat transfer fluid, and can be considered as one factor in the decision making process for fluid selection. The accuracy of the results by this test method depend very strongly on how closely the test conditions are followed.

The procedure is applicable to heat transfer fluids at temperatures both above and below their boiling points. It is applicable to fluids with maximum bulk operating temperature between 500°F and 850°F. The procedure shall not be used to test a fluid above its critical temperature. In this test method the volatile decomposition products are in continuous contact with the fluid during the test. This test will not measure the thermal stability threshold (the temperature at which volatile oil fragments begin to form), but instead will indicate bulk fragmentation occurring for a specified temperature and testing period. Because potential decomposition and generation of high pressure gas may occur at temperatures above 500°F, this test method should not be used for aqueous fluids or other fluids which generate high pressure gas at these temperatures. The applicability of this test method to siloxane based heat transfer fluids has not been determined. DIN Method 51528 covers a test method that is similar to this test method.

TEST SUMMARY

The test fluid is charged in a thermal stability test cell purged with nitrogen. The cell is tightly sealed to remove and preclude introduction of oxygen and water from the atmosphere. The fluid is heated in an oven at a given temperature and for a given period of time. The boiling range of the heated fluid is determined by gas chromatography, and is compared to the boiling range of pure unused fluid.

TEST PRECISION

No data are available at present regarding the repeatability or reproducibility of this test method. This procedure has no bias because the value of thermal stability is defined only in terms of this test method.

THERMAL STABILITY OF HYDRAULIC OILS: D 2070

EXPLANATION

Thermal stability characterizes physical and chemical property changes that may adversely affect an oil's lubricating performance. This test method evaluates the thermal stability of a hydraulic oil in the presence of copper and steel at 135°C. Rod colors are the evaluation criteria. Sludge values are reported for informational purposes. No correlation of this test with field service has been made.

TEST SUMMARY

A beaker containing the test oil, copper and iron rods are placed in an aluminum block in an electric gravity convection oven for 168 h at a temperature of 135°C. At the end of this time, the copper and steel rods are rated visually for discoloration using the Cincinnati Milacron color chart, and the oil is analyzed for the quantity of sludge.

TEST PRECISION

Analysis	Repeatability	Reproducibility
Copper Rod Color	1 unit	4 units
Steel Rod Color	1 unit	2 unit
Total Sludge	$1.04(X+1)$	$3.25(X+1)$

Where X is the mean value.

The bias of this test method has not been determined.

INSTABILITY OF MIDDLE DISTILLATE FUELS BY PORTABLE SPECTROPHOTOMETER: D 6748

EXPLANATION

Storage stability depends on complex interactions. It varies with feedstock type and source, and the processing used. The rate of degradation may not change uniformly with temperature. Chemical reactions may lead to a change in color followed by the formation of soluble gum and insoluble sediments which may overload the filters, and plug nozzles and injectors. This test method provides a rapid procedure using a portable apparatus and allows the test to be carried out on site or in the laboratory to give a result within 35 min. The potential beneficial effects of stability additives in fuels may not be recognized by this test method. Therefore, the actual storage stability of middle distillate fuels with stability additives may not be correctly indicated by these test results. The unstable reactive products (phenalenes and phenalenones) detected by this test method may be present in fuels containing catalytically cracked or straight run materials and can affect the potential instability of the fuel. If this test method is used by any party for a rapid assessment of stability, it is the responsibilities of the parties involved to decide whether or not this procedure yields meaningful results.

This test method rapidly determines phenalenes and phenalenones in middle distillate fuels, including marine, automotive, heating, and gas turbine fuels such as those specified in Specifications D 396, D 975, D 2069, and D 2880. This test method is applicable to both dyed and undyed fuels at all points in the distribution chain from refinery to end-user. It is not applicable to fuels containing residual oil. This test method is suitable for testing samples with a relative absorbance of up to 5.00 absorbance units.

TEST SUMMARY

A 5 mL volume of middle distillate fuel is mixed with an equal volume of an immiscible reagent solution (a proprietary methanol solvent containing a Lowry-Bronsted acid). A second reagent solution (proprietary solution containing potassium permanganate and an oxidizing agent) is then added, the new blend mixed and allowed to settle for 30 min for two phases to separate. The top darker reagent layer is then placed in the portable spectrophotometer and the relative absorbance of near infrared and visible light, at fixed single wavelengths is measured instantaneously and automatically, and reported in absorbance units.

When mixed with oil samples containing phenalenes, the second oxidizing reagent oxidizes the phenalenes to phenalones which are subsequently converted to colored indolphenalene salts by the acidic first reagent. The spectrophotometer measures the absorbance of light caused by the colored salts while eliminating the effect of the initial color of the fuel. The light absorbance is a measure of the concentration of the colored salts that are formed.

TEST PRECISION

Repeatability:	0.0876(x+0.2)AU
Reproducibility:	0.1799(x+0.2)AU

Where x is the average of results being compared.

Since there is no accepted reference material suitable for determining the bias of the procedures in this method, bias cannot be determined.

THERMAL STABILITY OF SOLID FILM LUBRICANTS: D 2511

EXPLANATION

Solid lubricant coatings are applied to surfaces that are exposed to heat and cold to such a degree that in many cases liquid lubricants are not practical. Adherence under these conditions is mandatory to preserve the bearing surfaces during sliding motion. This test method covers the measurement of the resistance of dry solid film lubricants to deterioration when subjected to temperature extremes.

TEST SUMMARY

A steel panel having the solid film lubricant deposited on one surface is subjected to 260°C(500°F) heat followed by immediate exposure to −54°C(−65°F), each for a period of 3 h. The solid film is then examined for cracking, flaking, blistering, or other evidence of thermal instability.

TEST PRECISION

This is a pass-fail test, and the normal precision or bias statements are not applicable.

THERMAL STABILITY OF WAY LUBRICANTS: D 6203

EXPLANATION

Thermal stability characterizes physical and chemical property changes, which may adversely affect an oil's lubricating performance. This test method evaluates the thermal stability of hydrocarbon based way lubricant in the presence of copper and steel rods at 100°C. Deposits and rod colors are the evaluation criteria. No correlation of this test to field service has been made. This test method is intended for use in qualifying a way lubricant, rather than for quality control or condition monitoring purposes.

TEST SUMMARY

A beaker containing the test oil, and copper and steel rods are placed in a gravity convection electric oven for 24 h at a temperature of 100°C. At the end of this time, the copper and steel rods are visually rated for discoloration using the Cincinnati Milacron Color chart and the beaker is visually evaluated for deposits.

TEST PRECISION

The precision and bias of this test method are under study.

TOLUENE INSOLUBLES IN TAR AND PITCH: D 4072 and D 4312

EXPLANATION

These test methods are used for evaluating and characterizing tars and pitches and as one element in establishing the uniformity of shipments or sources of supply. Since these test methods are empirical, strict adherence to all details of the procedures is necessary.

TEST SUMMARY

The sample is digested at 95°C for 25 min, then extracted with hot toluene in an alundum thimble. The extraction time is 18 h in Test Method D 4072, and 3 h in Test Method 4312. The insoluble matter is dried and weighed.

TEST PRECISION

Test Method	Repeatability	Reproducibility
D 4072	0.9	2.0
D 4312	1.0	3.0

Toluene insolubles determined by Test Method D 4312 are higher by an average of about 1 percentage point compared to the value obtained by Test Method D 4072.

TORQUE, LOW TEMPERATURE OF BALL BEARING GREASE: D 1478

EXPLANATION

This test method covers the determination of the extent to which a grease retards the rotation of a slow speed ball bearing by measuring starting and running torques at low temperatures (<–20°C or 0°F). This test method has proved helpful in selecting greases for low powered mechanisms, such as instrument bearings used in aerospace applications. Test Method D 4693 (see next) may be better suited for applications using larger bearings or greater loads. These two test methods may not give the same torque values because the apparatus and test bearings are different.

TEST SUMMARY

A No. 6204 open ball bearing is packed completely full of grease and cleaned off flush with the sides. The bearing remains stationary while ambient temperature is lowered to the test temperature and held there for 2 h. At the end of this time, the inner ring of the ball bearing is rotated at 1 ± 0.05 r/min while the restraining force on the outer ring is measured. Torque is measured by multiplying the restraining force by the radius of the bearing housing. Both starting torque and after 60 min of rotation are determined.

TEST PRECISION

Analysis	Repeatability	Reproducibility
Starting Torque	34 %	79 %
Torque after 60 min	78 %	132 %

The values are percent of the mean.

This test method has no bias.

TORQUE, LOW TEMPERATURE OF GREASE LUBRICATED WHEEL BEARINGS: D 4693

EXPLANATION

This test method determines the extent to which a test grease retards the rotation of a specially manufactured, spring loaded, automotive type wheel bearing assembly when subjected to low temperatures. Torque values, calculated from restraining force determinations are a measure of the viscous resistance of the grease. This test is used for specification purposes and correlates with its precursor which has been used to predict the performance of greases in automotive wheel bearings in low temperature service.

TEST SUMMARY

A freshly stirred and worked sample of grease is packed into the bearings of a specially manufactured, automotive type spindle bearings hub assembly. The assembly is heated and then cold soaked at −40°C. The spindle is rotated at 1 r/min and the torque required to prevent rotation of the hub is measured at 60 s.

TEST PRECISION

Repeatability:	0.22 M
Reproducibility:	0.55 M

Where M is the average of two results.

This test method has no bias.

TOTAL INHIBITOR CONTENT OF LIGHT HYDROCARBONS:
D 1157

EXPLANATION

p-tertiary-butyl catechol (TBC) is commonly added to commercial butadiene in amounts of 50 to 250 mg/kg as an oxidation inhibitor. This test method is suitable for use by both producers and users of butadiene. In general, all phenols and their quinone oxidation products are included in the calculated catechol content. Small amounts of polymer do not interfere. The test method is applicable over the range of TBC from 50 to 500 mg/kg.

TEST SUMMARY

The catechol is separated from the butadiene by evaporation. The residue is dissolved in water and an excess of ferric chloride is added. The intensity of the yellow-colored complex is compared in a photoelectric colorimeter with that produced by known concentrations of catechol.

TEST PRECISION

In the TBC concentration range of 50 to 500 mg/kg, this test method has a repeatability of 10 and a reproducibility of 20.

The bias of this test method has not been determined.

TRANSITION TEMPERATURES OF PETROLEUM WAXES BY DSC:
D 4419

EXPLANATION

Since petroleum wax is a mixture of hydrocarbons with different molecular weights, its transitions occur over a temperature range. This test method measures the transition temperatures of petroleum waxes, including microcrystalline waxes, by differential scanning calorimetry (DSC). These transitions may occur as a solid-solid transition or as a solid-liquid transition. The former is related to the properties of the solid, that is, hardness and blocking temperature. The second is the highest temperature transition associated with complete melting; it can guide the choice of wax storage and application temperatures.

The normal operating temperature range is from 15–150°C. Test Method D 87 also measures energy transfer between wax and a standard environment. The highest temperature DSC transition may differ from the melting point because the two methods approach the solid/liquid phase transition from different directions. The DSC method cannot differentiate between solid-liquid and solid-solid transitions. Such information must be predetermined by other techniques. In case of blends, the lowest temperature transition may be envelopes of both solid-liquid and solid-solid transitions.

TEST SUMMARY

Separate samples of petroleum wax and a reference material or blank (empty sample container) are heated at a controlled rate in an inert atmosphere. A sensor continuously monitors the difference in heat flow to the two samples. The DSC curve is a record of this difference versus temperature. A transition in the wax involves the absorption of energy relative to the reference, resulting in an endothermic peak in the DSC curve. While the transition occurs over the temperature range spanned by the base of the peak, the temperature associated with the peak apex is designated the nominal transition temperature.

Since the sample size taken for analysis is only 10 mg, it is particularly important that the test specimen be homogenous and representative of the bulk material. Intimate thermal contact, sample-to-pan and pan-to-sensor, is essential to obtain accurate and reproducible results. The heating rate must be the specified 10±1°C/min. Faster or slower rates will produce a different transition temperature and transition peak width.

TEST PRECISION

Test Parameter	Repeatability		Reproducibility	
	°C	°F	°C	°F
Solid-Liquid Transition Temperature				
-Apex, T_{2A}	0.8	1.4	3.5	6.3
-End, T_{2E}	1.0	1.8	6.1	11.0
Solid-Solid Transition Temperature				
-Apex T_{1A}	1.2	2.2	2.3	4.1
-End T_{1E}	1.4	2.5	11.2	20.2

The procedure in this test method has no bias because the value of the transition temperature can be defined only in terms of this test method.

ULTRAVIOLET (UV) ABSORBANCE OF PETROLEUM PRODUCTS:
D 2008

EXPLANATION

The absorbance of liquids and the absorptivity of liquids and solids at specified wavelengths in the ultraviolet region (220 to 400 nm) are useful in characterizing petroleum products. Examples of the application of this test method are the determination of the absorbance of white mineral oil, the absorptivity of refined petroleum wax, and the absorptivity of USP petrolatum.

TEST SUMMARY

The UV absorbance of a liquid is determined by measuring the absorption spectrum of the undiluted liquid in a cell of known path length under specified conditions. The UV absorptivity of a solid or a liquid is determined by measuring the absorbance, at specified wavelengths, of a solution of the liquid or solid at known concentration in a cell of known path length.

TEST PRECISION

Product	Measurement	Repeatability	Reproducibility
White Mineral Oil	Wavelength, nm		
	275	0.008	0.053
	295 to 299	0.019	0.071
	300 to 400	0.014	0.080
Refined Petroleum	0.15	0.02	0.05
Wax Absorptivity	0.30	0.02	0.05
Petrolatum	Absorptivity 0.5	0.01	0.05
Extender Oils	Absorptivity	7 %	9 %

The bias for this test method has not been determined.

UNSULFONATED RESIDUE OF OILS: D 483

EXPLANATION

This test method is useful for distinguishing between oils that are adaptable to various types of spraying application, with a higher sulfonated oil being required for leaf spraying as compared to dormant vegetation application. Since the relationship between unsulfonated residue and the actual composition of the oil is not known, this test method should be applied only for measuring the degree of refinement and not for the determination of aromatics or olefins, or both.

TEST SUMMARY

A measured volume of the sample is shaken with 98.61 % sulfuric acid at 100°C in a Babcock bottle, shaking mechanically for 10 s at 10 min intervals. The volume not absorbed by the acid is a measure of the unsulfonated residue in the sample.

TEST PRECISION

In the 79 to 97 % unsulfonated range, this test method has a repeatability of 0.5 and a reproducibility of 2.0.

The bias of this test method is not known.

VANADIUM IN HEAVY FUEL OIL: D 1548

(Equivalent Test Method: AFNOR M07-027)

EXPLANATION

Vanadium in fuels can form low melting compounds that are severely corrosive to metal parts. This test method covers the determination of 0 to 350 mg/kg of vanadium in heavy fuel oils, and is widely cited in commercial specifications and contracts for marine and power plants Number 6 oils. Other elements commonly found in fuel oils do not interfere.

TEST SUMMARY

The fuel oil is heated with concentrated sulfuric acid and reduced to acid-free coke and then dry ashed at 525°C. Mixed nitric and sulfuric acids are then added to the residue and evaporated to the white fumes of sulfuric acid. The residue is dissolved in water, phosphoric acid and sodium tungstate are added, and vanadium is determined by measuring the absorbance of its phosphotungstovanadic acid complex at 436 nm.

TEST PRECISION

Vanadium, mg/kg	Repeatability	Reproducibility
0 to 2	0.3 ppm	
0 to 6		50 %
2 to 350	10 %	
6 to 350		17 %

VAPOR LIQUID RATIO OF FUELS: D 2533

EXPLANATION

The tendency of a fuel to vaporize in common automobile fuel systems is indicated by the vapor-liquid ratio of that fuel at conditions approximating those in critical parts of the fuel systems. Dry glycerol can be used as the containing liquid for nonoxygenated fuels. Mercury can be used as the containing liquid with both oxygenated and nonoxygenated fuels.

TEST SUMMARY

A measured volume of liquid fuel at 32 to 40°F is introduced through a rubber septum into a glycerol or mercury filled buret. The charged buret is placed in a temperature controlled water bath. The volume of vapor in equilibrium with liquid fuel is measured at the desired temperature or temperatures and the specified pressure, usually 760 mm Hg. The vapor-liquid ratio is then calculated.

TEST PRECISION

Medium	Repeatability	Reproducibility
Glycerol	1.0°C(1.8°F)	1.3°C(2.3°F)
Mercury	1.4°C(2.5°F)	1.6°C(3.0°F)

There is no bias between this test method and Test Method D 5188 (see next).

VAPOR LIQUID RATIO TEMPERATURE OF FUELS: D 5188

EXPLANATION

The tendency of a fuel to vaporize in automotive engine fuel systems is indicated by the vapor-liquid ratio of the fuel. Automotive fuel specifications generally include this ratio temperature. This test method is applicable to samples for which the determined temperature is between 36 and 80°C, and the vapor-liquid ratio is between 8:1 and 75:1. When the ratio is 20:1, the result is intended to be comparable to the results obtained by Test Method D 2533. This test may also be applicable at pressures other than 1 atmosphere, but the stated precision may not apply.

TEST SUMMARY

This test method determines the temperature at which the vapor formed from a selected volume of volatile petroleum product saturated with air at 0 to 1°C produces a pressure of 1 atmosphere in an evacuated chamber of fixed volume. A known volume of chilled, air-saturated sample is introduced into an evacuated, thermostatically controlled test chamber of known volume. The sample volume is calculated to give the desired liquid-vapor ratio for the chamber volume in use. After injection, the chamber temperature is adjusted until a stable chamber pressure of 101.3 kPa is achieved.

TEST PRECISION

Repeatability:	0.6°C(1.1°F)
Reproducibility:	0.9°C (1.6°F)

Bias has not been determined.

VAPOR PRESSURE OF CRUDE OIL: D 6377

EXPLANATION

Vapor pressure of crude oil at various vapor-liquid ratios is an important physical property for shipping and storage. This test method covers the use of automated vapor pressure instruments to determine the vapor pressure of crude oils at temperatures between 5 and 80°C for vapor-liquid ratios from 4:1 to 0.02:1 and pressures from 7 to 500 kPa. This test method allows the determination of $VPCR_x$ samples having pour points above 0°C.

The vapor pressure determined by this test method at a V/L of 4:1 of crude oil at 37.8°C can be related to the vapor pressure value determined on the same material when tested by the Test Method D 323.

TEST SUMMARY

Employing a measuring chamber with a built-in piston, a sample of known volume is drawn from a pressurized sampling system (floating piston cylinder) into the temperature controlled chamber at 20°C or higher. After sealing the chamber, the volume is expanded by moving the piston until the final volume produces the desired vapor-liquid ratio value. The temperature of the measuring chamber is then regulated to the measuring temperature. After temperature and pressure equilibrium, the measured pressure is recorded as the $VPCR_x$ of the sample. The test specimen is mixed during the measuring procedure by shaking the measuring chamber to achieve pressure equilibrium in a reasonable time between 5 and 30 min.

TEST PRECISION

Repeatability:
  V/L = 4 and 37.8°C:
    repeatability = 0.15 VPCR4 (37.8°C)
  V/L = 0.1 and 37.8°C:
    repeatability = 0.055 VPCR 0.1 (37.8°C)
  V/L = 0.2 and 37.8°C:
    repeatability = 0.065 VPCR0.02 (37.8°C)

Reproducibility is being determined. Bias is not known.

VAPOR PRESSURE OF GASOLINE AND BLENDS: D 4953

EXPLANATION

Vapor pressure is an important physical property of liquid spark-ignition engine fuels. It provides an indication of how a fuel will perform under different operating conditions, such as whether it will cause vapor lock at high ambient temperature or at high altitude, or will provide easy starting at low ambient temperature. Petroleum product specifications generally include vapor pressure limits to ensure products of suitable volatility performance. Vapor pressure of fuels is regulated by various government agencies.

This test method is a modification of Reid vapor pressure method Test Method D 323 (included here). This test method is applicable to gasolines and gasoline-oxygenate blends with a vapor pressure range of 35 to 100 kPa.

TEST SUMMARY

This test method provides two procedures for determining vapor pressure. The liquid chamber of the vapor pressure apparatus is filled with chilled sample and connected to the vapor chamber at 100°F. The apparatus is immersed in a bath of 100°F until a constant pressure is observed. The pressure reading, suitably corrected, is reported as the vapor pressure.

In Procedure A the same apparatus and essentially the same procedure as in Test Method D 323 is utilized with the exception that the interior surfaces of the liquid and vapor chambers are maintained completely free of water. Procedure B utilizes a semiautomatic apparatus with the liquid and vapor chambers identical in volume to those in Procedure A. The apparatus is suspended in a horizontal bath and rotated while attaining equilibrium. Either a Bourdon gage or pressure transducer can be used with this procedure. The interior surfaces of the liquid and vapor chambers are maintained free of water.

TEST PRECISION

Procedure	Repeatability	Reproducibility
A	3.65 kPa	5.52 kPa
B: Gage	4.00	5.38
B: Transducer (Herzog)	2.14	2.90
B: Transducer (Precision Scientific)	3.58	4.27

Bias of this test method has not been determined.

VAPOR PRESSURE OF LPG (EXPANSION METHOD): D 6897

EXPLANATION

Information on the vapor pressure of LPG is pertinent to selection of properly designed storage vessels, shipping containers, and customer utilization equipment to ensure safe handling of these products. It is important for safety reasons that the maximum operating design pressure of storage, handling, and fuel systems will not be exceeded under normal operating temperature conditions. For LPG, vapor pressure can be considered a semi-quantitative measure of the amount of the most volatile material present in the product. This test method uses a small sample volume and excludes any manual handling of a measuring chamber under high pressure. It covers product temperatures of 37.8°C, vapor to liquid ratio of 0.5:1, and pressures from 200–1550 kPa on a sample volume of 3.33 mL. This test method is applicable to the determination of vapor pressures of LPG products at temperatures from 37.8–70°C, vapor to liquid ratios of 0.1:1 to 4:1 and pressures up to 3500 kPa. This test method is not intended to determine the true vapor pressure of LPG samples, but rather determine the vapor pressure at the conditions mentioned above, just like the Test Method D 1267. Thus, this test method will not measure the full contribution from any dissolved gases such as nitrogen or helium if they are present. The contribution of light gases to the measured vapor pressure is highly dependent on the test temperature, type of gas, and V/L ratio of the test.

TEST SUMMARY

Employing a measuring chamber with a built-in piston, the chamber is rinsed three times with a portion of the sample, which is then discarded. A sample of defined volume is drawn from a pressurized sampling system into the temperature controlled chamber at 5°C by moving the piston into the filling position. After sealing the chamber, the volume is expanded by moving the piston until the final volume produces the desired vapor to liquid ratio of 0.5:1. The temperature of the measuring chamber is then regulated to the test temperature of interest, such as 37.8°C. The observed total pressure at equilibrium is corrected relative to 101.3 kPa and reported as the LPG vapor pressure at the selected test temperature.

TEST PRECISION

Repeatability:	7.4 kPa(1.1 psi)
Reproducibility:	Not known at present

Relative bias against Test Method D 1267 is not yet known.

VAPOR PRESSURE OF PETROLEUM PRODUCTS:
D 5190

EXPLANATION

See Test Method D 4953. This test method is applicable to air-containing, volatile petroleum products with boiling points above 0°C that exert a vapor pressure between 7 and 172 kPa at 100°F at a vapor-to-liquid ratio of 4:1. This test method is suitable for testing oxygenate containing gasolines. Using a correlation equation, this test method can calculate a dry vapor pressure equivalent which very closely approximates that obtained by Test Method D 4953.

TEST SUMMARY

The chilled sample cup of the automatic vapor pressure instrument is filled with chilled sample and is coupled to the instrument inlet fitting. The sample is then automatically forced from the sample chamber to the expansion chamber where it is held until thermal equilibrium at 100°F is reached. In this process the sample is expanded to five times its volume (4:1 vapor-to-liquid ratio). The vapor pressure is measured by a pressure transducer. The measured vapor pressure is automatically converted to a dry vapor pressure equivalent value by the instrument.

TEST PRECISION

Repeatability:	2.48 kPa
Reproducibility:	3.45 kPa

Bias has not been determined.

VAPOR PRESSURE OF PETROLEUM PRODUCTS:
D 5191
(Equivalent Test Methods: IP 394 and AFNOR M07-079)

EXPLANATION

See Test Method D 4953. This test method is, however, more precise, uses a small sample size (1 to 10 mL), and requires about 7 min to complete the test. See Explanation under Test Method D 5190.

TEST SUMMARY

See Test Method D 5190.

TEST PRECISION

Repeatability:	0.00807(DVPE+124)
Reproducibility:	0.0161(DVPE+124)

Bias has not been determined.

VAPOR PRESSURE OF PETROLEUM PRODUCTS: D 5482

EXPLANATION

See Test Method D 4953.

TEST SUMMARY

See Test Method D 5190.

TEST PRECISION

Instrument	Repeatability	Reproducibility
Herzog model SC 970	1.31 kPa	2.69 kPa
ABB model 4100	1.79	4.14

The bias of this test method has not been determined.

VAPOR PRESSURE REID VAPOR PRESSURE OF PETROLEUM PRODUCTS:

D 323

(Equivalent Test Methods: IP 69, ISO 3007, DIN 51754, JIS K 2258, and AFNOR M41-007)

EXPLANATION

See Test Method D 4953. This test method measures vapor pressure of gasoline, volatile crude oil, and other volatile petroleum products. Procedure A is applicable to samples with a vapor pressure of less than 180 kPa. Procedure B may also be applicable to these products, but only gasoline samples were included in the precision study. Neither procedure is applicable to LPG or fuels containing oxygenated compounds other than MTBE. Procedure C is for materials with a vapor pressure of greater than 180 kPa. Procedure D is for aviation gasoline with a vapor pressure of approximately 50 kPa.

Reid vapor pressure differs from the true vapor pressure of the sample due to some small sample vaporization and the presence of water vapor and air in the confined space.

TEST SUMMARY

See Test Method D 4953. All four procedures utilize liquid and vapor chambers of the same internal volume. Procedure B utilizes a semiautomatic apparatus immersed in horizontal bath and rotated while attaining equilibrium. Either a Bourdon gage or pressure transducer may be used with this procedure. Procedure C utilizes a liquid chamber with two valved openings. Procedure D requires more stringent limits on the ratio of the liquid and vapor chambers.

TEST PRECISION[1]

See the table below for precision estimates.

[1] This table is from the section on precision and bias from Test Method D 323.

	Range		Repeatability		Reproducibility	
Procedure	kPa	psi	kPa	psi	kPa	psi
A Gasoline	35–100	5–15	3.2	0.46	5.2	0.75
B Gasoline	35–100	5–15	1.2	0.17	4.5	0.66
A	0–35	0–5	0.7	0.10	2.4	0.35
A	110–180	16–26	2.1	0.3	2.8	0.4
C	>180	>26	2.8	0.4	4.9	0.7
D Aviation Gasoline	50	7	0.7	0.1	1.0	0.15

VAPOR PRESSURE OF PETROLEUM PRODUCTS: D 6378

EXPLANATION

Vapor pressure is a very important physical property of volatile liquids for shipping and storage. The vapor pressure of gasoline and gasoline-oxygenate blends is regulated by various government agencies. Specifications for volatile petroleum products generally include vapor pressure limits to ensure products of suitable volatility performance.

This test method covers the use of automated instruments to determine the vapor pressure exerted in vacuum by volatile, liquid petroleum products, hydrocarbons, and hydrocarbon-oxygenate mixtures. This test method is suitable for samples with boiling points above 0°C that exert a vapor pressure between 7 and 150 kPa at 37.8°C at a vapor-to-liquid ratio of 4:1. This test method may be suitable for measurement of volatile petroleum liquids under conditions different than these but the precision statement may not be applicable. This test method can be applied in on-line applications in which an air saturation procedure prior to the measurement cannot be performed. The TP_x determined by this test method can be correlated to the dry vapor pressure equivalent value determined by Test Method D 5191.

TEST SUMMARY

Employing a measuring chamber with a built-in piston, a sample of known volume is drawn into the temperature controlled chamber at 20°C or higher. After sealing the chamber, an expansion is performed in three steps to a final volume of $(X+1)$ times that of the test specimen. After each expansion the TP_x is measured. The PPA and the solubility of air in the specimen are calculated from three resulting pressures. The temperature of the chamber is then increased to a specified value, and the TP_x is determined. The vapor pressure is calculated by subtracting the PPA in the liquid, which has been gas corrected for the temperature, from TP_x.

TEST PRECISION

Repeatability:	0.50 kPa(0.07 psi)
Reproducibility:	1.63 kPa(0.22 psi)

Relative bias is significant relative to Test Method D 5191.

VISCOSITY, APPARENT OF HOT MELT ADHESIVES: D 3236

EXPLANATION

This test method covers the determination of the apparent viscosity of hot melt adhesives and coating materials compounded with additives and having apparent viscosities up to 200 000 mPa.s at temperatures up to 175°C. It is believed that apparent viscosity determined by this test method is related to flow performance in application machinery operating under conditions of low shear rate. The results of this test may not correlate well with end use applications where high shear rates are encountered. This test method may be adaptable to viscosities higher than the present 200 000 mPa.s limit and temperatures above 175°C. However, precision of this case has not been studied.

TEST SUMMARY

A sample of molten material is maintained in a thermally controlled sample chamber. Apparent viscosity is determined under temperature equilibrium conditions using a precision rotating spindle type viscometer. Data obtained at several temperatures can be plotted on appropriate semi-log graph paper and apparent viscosity at intermediate temperatures can be estimated.

TEST PRECISION

Repeatability:	8.8 % of the mean
Reproducibility:	25.4 % of the mean

There is no bias.

VISCOSITY, APPARENT OF PETROLEUM WAXES: D 2669

EXPLANATION

See explanation under Test Method D 3236. This test method is applicable to fluid hot melts having apparent viscosities up to 20 Pa·s at temperatures up to 175°C.

TEST SUMMARY

Approximately 800 g of the sample is melted on a hot plate or in an oven. An 800 mL beaker, which is jacketed with an electric heating mantle, is filled with the melted sample to a level of about 1 in. from the top. The viscometer, with attached spindle and guard, is properly positioned. Stirring is begun and continued while the temperature of the sample is brought to slightly above the highest desired test temperature. Heating is discontinued but stirring is maintained until the sample cools to the chosen temperature. At this point the apparent viscosity is determined. Additional determinations are made as the sample cools. Results of temperature and apparent viscosity are plotted on a semilog paper, and values at any particular temperature are determined from the curve.

TEST PRECISION[1]

See the precision results as follows:

High-Viscosity Sample, MI-65-20:

58 weight % of a 68°C(155°F) melting point wax

42 weight % of an ethylene-vinyl acetate copolymer containing 27 to 29 % vinyl acetate and having a melt index of from 12 to 18

Viscosity, mPa·s	Temperature, °C	(°F)	Repeatability, mPa·s	Reproducibility, mPa·s
11 200	121	(250)	1900	2400
7500	134	(275)	1200	1700
5100	149	(300)	660	1500

Medium-Viscosity Sample, MI-65-21:

72 weight % of a 61°C(142°F) melting point wax

28 weight % of the same copolymer used in sample MI-65-20

Viscosity, mPa·s	Temperature, °C	(°F)	Repeatability, mPa·s	Reproducibility, mPa·s
1200	121	(250)	81	240
840	134	(275)	63	150
610	149	(300)	47	120

Low-Viscosity Sample, MI-65-22:

96.3 weight % of a 77°C(170°F) melting point microcrystalline wax

2.7 weight % of butyl rubber

Viscosity, mPa·s	Temperature, °C	(°F)	Repeatability, mPa·s	Reproducibility, mPa·s
68	121	(250)	15	32
52	134	(275)	11	29
41	149	(300)	7.9	22

Low-Viscosity Sample, MI-65-23:

80 weight % of a 68°C(154°F) melting point wax

20 weight % of a 5000 molecular weight polyethylene having a melting point from 107 to 111°C (224 to 232°F), a specific gravity of 0.92 and a typical viscosity at 140°C of 4 Pa·s.

Viscosity, mPa·s	Temperature, °C	(°F)	Repeatability, mPa·s	Reproducibility, mPa·s
25	121	(250)	1.9	4.4
20	134	(275)	1.2	4.0
16	149	(300)	1.5	3.8

[1] This table is from the precision and bias section of Test Method D 2669.

YIELD STRESS AND APPARENT VISCOSITY OF USED ENGINE OILS AT LOW TEMPERATURE:
D 6896

EXPLANATION

When an engine oil is cooled, the rate and duration of cooling can affect its yield stress and viscosity. In this test method, used engine oil is slowly cooled through a temperature range where wax crystallization is known to occur, followed by relatively rapid cooling to the final test temperature. As in other low temperature rheological tests such as Test Methods D 3829, D 4684, and D 5133, a preheating condition is required that all residual waxes are solubilized in the oil prior to the cooldown (i.e., remove thermal memory). However, it is also known that highly sooted diesel engine oils can experience a soot agglomerization phenomenon when heated under quiescent conditions. The current method uses a separate preheat and agitation step to break up any soot agglomerization that may have occurred prior to cooldown. The viscosity of highly sooted diesel engine oils as measured in this test method have been correlated to pressurization times in a motored engine test.

This test method covers the measurement of the yield stress and viscosity of engine oils after cooling at controlled rates over a period of 43 or 45 hours to a final test temperature of −20 or −25°C. The viscosity measurements are made at a shear stress of 525 Pa over a shear rate of 0.4 to 15 s^{-1}. This test method is suitable for measurement of viscosities ranging from 400 mPa·s to >400,000 mPa·s, and is suitable for yield stress measurements of 7 Pa to >350 Pa. The test method is applicable for used diesel oils. Other used or unused engine oils or other petroleum products have not been tested by this procedure.

TEST SUMMARY

A used engine oil sample is heated at 80°C and then vigorously agitated. The sample is then cooled at a programmed cooling rate to a final test temperature. A low torque is applied to the rotor shaft to measure the yield stress. A higher torque is then applied to determine the apparent viscosity of the sample.

TEST PRECISION

Parameter	Temperature, °C	Repeatability	Reproducibility
Yield Stress	−20	$0.543(X+1)$ Pa	$0.926(X+1)$ Pa
	−25	$0.504(X+1)$ Pa	$0.773(X+1)$ Pa
Apparent Viscosity	−20	$0.0879\ X$ %	$0.186\ X$ %
	−25	$0.0616\ X$ %	$0.209\ X$ %

Where X is the mean of the results.

Since there is no accepted reference material suitable for determining the bias for this test method, no statement on bias is being made.

VISCOSITY BROOKFIELD VISCOSITY:
D 2983
(Equivalent Test Methods: IP 267, ISO 9262, and AFNOR T42-011)

GENERAL

Many petroleum products are used as lubricants for bearings, gears, compressor cylinders, hydraulic equipment, etc. The proper operation of the equipment depends upon the proper kinematic viscosity or viscosity (sometimes called dynamic viscosity) of the liquid. Thus, the accurate measurement of kinematic viscosity and viscosity is essential to many product specifications.

The kinematic viscosity of many petroleum fuels is important for their proper use, for example, flow of fuels through pipe lines, injection nozzles and orifices, and the determination of the temperature range for proper operation of the fuel in burners.

EXPLANATION

The low-temperature, low-shear-rate viscosity of gear oils, automatic transmission fluids, torque and tractor fluids, and industrial and automotive hydraulic oils are frequently specified by Brookfield viscosities. This test method describes the use of the Brookfield viscometer for the determination of the low-shear-rate viscosity of automotive fluid lubricants in the temperature range from −5 to −40°C. The viscosity range is 500 to 1 000 000 cP (mPa·s). Brookfield viscosity is expressed in centipoises (1 cP=1 mPa·s). Its value may vary with the spindle speed (shear rate) of the Brookfield viscometer because many automotive fluid lubricants are non-Newtonian at low temperatures.

TEST SUMMARY

A lubricant fluid sample is cooled in an air bath at test temperature for 16 h. It is carried in an insulated container to a nearby Brookfield viscometer where its Brookfield viscosity is measured at any test temperature in the range from −5 to −40°C.

TEST PRECISION

Repeatability:	$3.79(X/1000)^{1.7}$
Reproducibility:	$11.31(X/1000)^{1.7}$

Where X is the mean result.

VISCOSITY SCANNING BROOKFIELD VISCOSITY: D 5133

EXPLANATION

The low-temperature, low-shear, rheological behavior of an engine oil plays a major role in determining whether the oil will flow to the inlet screen and then to the oil pump in sufficient quantities that the oil pump will be able to supply lubricant to the critical areas of the engine shortly after starting. Without such supply, engine failure can occur within a few minutes. This test method permits the relatively rapid determination of the suitability of an engine oil to flow to the pump inlet screen at low temperatures. Critical pumpability temperatures can be determined readily without deliberate choice of single or multiple test temperatures since all engine oils are tested in the same scanning manner over a −5 to −45°C temperature range. Applicability to petroleum products other than engine oils has not been determined. The three pumpability tests—Test Methods D 3829, D 4684, and this one, Test Method D 5133—use different shear stress and shear rates, and this can lead to differences in measured viscosity by these test methods among some test oils.

TEST SUMMARY

An oil, preheated to a specified temperature for a specified minimum time, is placed in a special test cell that is in turn immersed in temperature programmable liquid bath. The test cell is calibrated with calibration oil over the range of viscosities and temperatures of interest. The viscosity of the sample oil is then measured continuously over the chosen temperature range using a rotor turning within the test cell as the temperature is initially lowered, at 1°C/h, through the temperature range of interest. The torque required to shear the oil over the temperature range spanned during the test is continuously recorded. This torque record is used to calculate the oil viscosity. The critical pumpability temperature is stated to be that temperature at which the viscosity exceeds the value of critical pumpability viscosity.

TEST PRECISION

Viscosity, cP	Repeatability	Reproducibility
10 000	6.65	1.42
15 000	0.93	1.92
20 000	1.00	2.08
25 000	1.09	2.23
30 000	0.88	3.25
35 000	0.86	1.90
40 000	0.80	1.99

The bias of this test method has not been determined.

KINEMATIC VISCOSITY OF LIQUIDS BY AUTOMATED HOUILLON VISCOMETER:

D 7279

EXPLANATION

See under D 445 kinematic viscosity test method description. The Houillon viscometer tube method offers automated determination of kinematic viscosity. A typically a sample volume of less than 1 mL is required for the analysis. The method is applicable to both fresh and used lubricating oils. The range of kinematic viscosity covered by this test method is from 0.2 to 1000 mm^2/s in the temperature range between 20° and 150°C.

TEST SUMMARY

The kinematic viscosity is determined by measuring the time taken for a sample to fill a calibrated volume at a given temperature. The specimen is injected into the apparatus and then flows into the viscometer tube which is equipped with two detection cells. The specimen reaches the test temperature of the viscometer bath and when the leading edge of the specimen passes in front of the first detection cell, the automated instrument starts the timing sequence. When the leading edge of the specimen passes in front of the second detection cell, the instrument stops timing the flow. The time interval thus measured allows the calculation of the kinematic viscosity using a viscometer constant determined earlier by calibration with certified viscosity reference standards.

TEST PRECISION

Temperature, °C	Repeatability	Reproducibility	Range, mm^2/s
40	0.68 %	3.0 %	6–17
100	1.6 %	5.6 %	25–150

Bias — Since there is no accepted reference standard suitable for this determination, no bias can be determined. However, based on interlaboratory studies conducted following relative bias statements can be made:

At 40°C D 445$_{\text{predicted}}$ = (Houillon viscosity result) $-$ 0.290

At 100°C D 445$_{\text{predicted}}$ = (Houillon viscosity result) $-$ 0.133

At 40°C D 7042$_{\text{predicted}}$ = 0.9952 (Houillon viscosity result)

There were not sufficient D 7042 results obtained at 100°C to calculate correlation between D 7042 and Houillon results at that temperature.

VISCOSITY AT HIGH SHEAR RATE BY TAPERED BEARING SIMULATOR VISCOMETER AT 100°C:

D 6616

EXPLANATION

Viscosity at the shear rate and temperature of this test method is thought to be particularly representative of bearing conditions in large medium speed reciprocating engines as well as automotive and heavy duty engines operating in this temperature regime. The importance of viscosity under these conditions has been stressed in railroad specifications. This test method covers the laboratory determination of the viscosity of engine oils at 100°C and 1.10^6 s^{-1} using the Tapered Bearing Simulator (TBS) viscometer.

This test method is similar to Test Method D 4683 which uses the same TBS viscometer to measure high shear viscosity at 150°C. The Newtonian calibration oils used to establish this test method range from approximately 5 to 12 mPa·s (cP0 at 100°C), and either the manual or automated protocol was used by each participant in developing the test precision. The viscosity range of the test method at this temperature is from 1 mPa·s to above 25 mPa·s, depending on the model of TBS used. The non-Newtonian reference oil used to establish the shear rate of 1.10^6 s^{-1} for this test method has a viscosity of approximately 10 mPa·s at 100°C. Application of this method to petroleum products other than engine oils has not been determined in preparing viscometric information for this test method.

TEST SUMMARY

A motor drives a tapered rotor closely fitted inside a matched tapered stator. Appropriate technique establishes operation of the viscometer to yield 1.10^6 s^{-1} at a temperature of 100°C at which point test oils are introduced into the gap between the spinning rotor and stationary stator. The rotor exhibits a reactive torque to the viscous resistance of each test oil and the value of this torque response is used to determine the apparent viscosity of the test oil.

TEST PRECISION

Repeatability:	1.2 % of the mean
Reproducibility:	3.5 % of the mean

There is no accepted reference materials suitable for determining the bias of this test method.

VISCOSITY INDEX: D 2270

(Equivalent Test Methods: IP 226, ISO 2909, and AFNOR T60-136)

EXPLANATION

The Viscosity Index is a widely used and accepted measure of the variation in kinematic viscosity due to changes in the temperature of a petroleum product between 40 and 100°C. Viscosity index is an arbitrary number used to characterize the variation of the kinematic viscosity of a petroleum product with temperature. For oils of similar kinematic viscosity, the higher the viscosity index the smaller is the effect of temperature on its kinematic viscosity. This practice specifies the procedures for calculating the viscosity index of petroleum products, such as lubricating oils, and related materials from their kinematic viscosities at 40 and 100°C.

TEST PRECISION

The calculation of viscosity index from kinematic viscosities at 40 and 100°C is exact, and no precision limits can be assigned to this calculation.

The accuracy of the calculated viscosity index is dependent only on the accuracy of the original viscosity determination. Test Method D 445 has a stated repeatability limit of 0.35 % and a reproducibility limit of 0.70 %.

The calculation of viscosity index from kinematic viscosities at 40 and 100°C is exact, and no bias can be assigned to this calculation.

VISCOSITY, KINEMATIC OF AIRCRAFT TURBINE LUBRICANTS:

D 2532

EXPLANATION

Aircraft turbine lubricants, upon standing at low temperatures for prolonged periods of time, may show an increase in kinematic viscosity. This increase may cause lubrication problems in aircraft engines. Thus, this test method is used to ensure that the kinematic viscosity does not exceed the maximum kinematic viscosity in certain specifications for aircraft turbine lubricants.

TEST SUMMARY

Kinematic viscosity is measured at low temperature in accordance with Test Method D 445, and at time intervals of 3 and 72 h.. This test was developed at −65°F, and has been used at −40°F. It may be used at other temperatures as agreed by the contracting parties.

TEST PRECISION

See the precision in the following figure.

The bias of this test method has not been established.

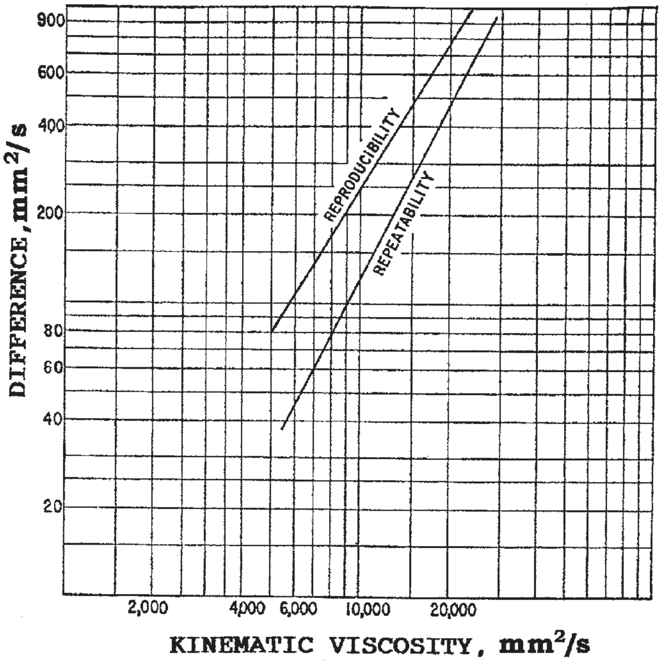

FIG. 20—*Repeatability and Reproducibility.*

VISCOSITY, KINEMATIC OF TRANSPARENT AND OPAQUE LIQUIDS:

D 445

(Equivalent Test Methods: IP 71-1, SO 3104, DIN 51562, JIS 2283 and AFNOR T60-100)

EXPLANATION

This test method covers the determination of the kinematic viscosity of liquid petroleum products, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity can be obtained by multiplying the measured kinematic viscosity by the density of the liquid. This test method is primarily intended for application to liquids for which the shear stress and shear rates are proportional. This test method also includes the determination of the kinematic viscosity of fuel oils which often exhibit non-Newtonian properties.

DEFINITIONS

Kinematic viscosity—a measure of the resistive flow on a fluid under gravity, the pressure head being proportional to the density, ρ, of the fluid: for gravity flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density, ρ. For any particular viscometer, the time of flow of a fixed volume of fluid is directly proportional to its kinematic viscosity, $\nu = \eta / \rho$, where η is the dynamic viscosity coefficient. The kinematic viscosity coefficient has the dimension L^2/T, where L is a length, and T is a time. The cgs unit of kinematic viscosity is one centimetre squared per second and is called one stokes (symbol St). The SI unit of kinematic viscosity is one metre squared per second and is equivalent to 10^4 ST. Frequently, the centistokes (symbol cSt) is used (1 cSt = 10^{-2} St = 1 mm^2/s).

TEST SUMMARY

The time is measured in seconds for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer.

TEST PRECISION[1]

[1] This table is from the section on precision in Test Method D 445.

	Determinability		Repeatability		Reproducibility	
Additives at 100°C	$0.00106Y^{1.1}$		$0.00192X^{1.1}$		$0.00862X^{1.1}$	
Base oils at 40 and 100°	$0.0020y$	(0.20 %)	$0.0011x$	(0.11 %)	$0.0065x$	(0.65 %)
Formulated oils at 40 and 100°C	$0.0013y$	(0.13 %)	$0.0026x$	(0.26 %)	$0.0076x$	(0.76 %)
Formulated oils at 150°C	$0.015y$	(1.5 %)	$0.0056x$	(0.56 %)	$0.018x$	(1.8 %)
Gas Oils at 40°C	$0.0013\ (Y+1)$		$0.0043\ (X+1)$		$0.0082\ (X+1)$	
Jet Fuels at −20°C	$0.0018\ Y$	(0.18 %)	$0.007\ X$	(0.7 %)	$0.019\ X$	(1.9 %)
Petroleum wax at 100°C	$0.0080y$	(0.80 %)	$0.0141x^{1.2}$		$0.0366x^{1.2}$	
Residual fuel oils at 80 and 100°C	$0.011^{(y+8)}$		$0.013^{(x+8)}$		$0.04^{(x+8)}$	
Residual fuel oils at 50°C	$0.017y$	(1.7 %)	$0.015x$	(1.5 %)	$0.74x$	(7.4 %)

Where y is the average of determinations being compared; x is the average of results being compared.

The precision for used oils has not been determined but is expected to be poorer than that for formulated oils. Because of the extreme variability of such used oils, it is not anticipated that the precision of used oils will be determined.

VISCOSITY, KINEMATIC OF VOLATILE AND REACTIVE LIQUIDS:

D 4486

EXPLANATION

Kinematic viscosity is a physical property that is of importance in the design of systems in which flowing liquids are used or handled. This test method is applicable to transparent Newtonian liquids that because of their reactivity, instability, or volatility cannot be used in conventional capillary kinematic viscometers. This test method is applicable up to 2 atm pressure and temperature range from −53 to +135°C.

TEST SUMMARY

The time is measured, in seconds, for a fixed volume of liquid to flow under gravity through the capillary of the viscometer under a reproducible driving head and at a closely controlled temperature. The kinematic viscosity is calculated from the measured flow time and the calibration constant of the viscometer.

TEST PRECISION

The precision and bias of this test method are expected to be substantially identical to that given in Test Method D 445 because the functional part of the viscometer used in this test method is identical to the Ubbelohde viscometer specified in Test Method D 445.

VISCOSITY, SAYBOLT:
D 88

EXPLANATION

This test method covers the empirical procedures for determining the Saybolt Universal or Saybolt Furol viscosities of petroleum products at specified temperature between 21 and 99°C (70 and 210°F).

Test Methods D 445 and D 2170 are preferred for the determination of kinematic viscosity. They require smaller samples and less time, and provide greater accuracy. Kinematic viscosities may be converted to Saybolt viscosities by use of the tables in Test Method D 2161. It is recommended that viscosity indexes be calculated from kinematic rather than Saybolt viscosities.

DEFINITIONS

Saybolt Universal viscosity—the corrected efflux time in seconds of 60 mL of sample flowing through a calibrated Universal orifice under specified conditions. The viscosity value is reported in Saybolt Universal seconds, abbreviated SUS, at a specified temperature.

Saybolt Furol viscosity—the corrected efflux time in seconds of 60 mL of sample flowing through a calibrated Furol orifice under specified conditions. The viscosity value is reported in Saybolt Furol seconds, abbreviated SFS, at a specified temperature.

Furol—an acronym of "*Fu*el and *r*oad *oil*s."

TEST SUMMARY

The efflux time in seconds of 60 mL of sample, flowing through a calibrated orifice, is measured under carefully controlled conditions. This time is corrected by an orifice factor and reported as the viscosity of the sample at that temperature.

TEST PRECISION

No precision or bias data are available at present.

LOW TEMPERATURE VISCOSITY OF DRIVE LINE LUBRICANTS IN A CONSTANT SHEER STRESS VISCOMETER:
D 6821

EXPLANATION

Viscosity of drive line lubricants at low temperature is critical for both gear lubrication and the circulation of the fluid in automatic transmissions. For gear oils, the issue is whether the fluid characteristics are such that the oil will flow into the channel dug out by the submerged gears as they begin rotating and relubricating them as they continue to rotate. For automatic transmission fluids, torque, and tractor fluids the issue is whether the fluid will flow into a pump and through the distribution system rapidly enough for the device to function. The low temperature performance of the drive line lubricant flow characteristics was originally evaluated by channel test. In this test, a pan was filled to a specified depth of approximately 2.5 cm and then cooled to test temperature. The test was performed by scraping a channel through the full depth of the fluid and across the length of the pan after it had soaked at test temperature for a specified time. The time it took the fluid to cover the channel was measured. The channel test was replaced by Test Method D 2983. The results of this Test Method D 6821 correlates with the viscometric measurements obtained in the Test Method D 2983 by multiplying the latter data with 0.941.

This test method is applicable to drive line lubricants (gear oils, automatic transmission fluids, etc.) with a constant shear stress viscometer at temperatures from −40 to 10°C after a prescribed preheat and controlled cooling to the final test temperature. The applicability of this test method to petroleum products other than drive line lubricants has not been determined.

TEST SUMMARY

A drive line fluid is preheated to 50°C for a specified time, and then cooled at a programmed rate to the final test temperature, and soaked at the final temperature for a defined period of time. At the completion of the soak time, the viscosity is measured by applying a prescribed torque and measuring rotational speed to determine the apparent viscosity of the sample.

TEST PRECISION

Repeatability:	10.3 % of the mean
Reproducibility:	17.2 % of the mean

There is no accepted reference material suitable for determining the bias of this test method, hence, no statement of bias can be made.

ROTATIONAL VISCOSITY OF HEAVY DUTY DIESEL DRAIN OILS AT 100°C:

D 6895

EXPLANATION

Rotational viscosity measurements allow the determination of the non-Newtonian, shear thinning property of drain oil. Rotational viscosity values can be compared at a shear rate of 100 s^{-1} by this test method. This test method covers the determination of the rotational viscosity and the shear thinning properties of heavy duty diesel engine drain oils at 100°C in the shear range of approximately 0.1–10 Pa and the viscosity range of approximately 12–35 mPa·s.

TEST SUMMARY

The sample is placed in a controlled stress or controlled shear rate rheometer/viscometer at 100°C. The sample is preheated at 10 s^{-1} for 30 s followed by heating at 100°C for 10 min. An increasing shear rate (approximately 10–300 s^{-1}) or shear stress (0.1–10 Pa) sweep is run followed by a decreasing sweep. The rotational viscosity for each step (increasing and decreasing) at 100 s^{-1} shear rate is interpolated from the viscosity versus shear rate data table. The rate index, as a measure of shear thinning, is calculated from a plot of In (shear stress) versus In (shear rate).

TEST PRECISION

The precision was found to be dependent on the mean value of the measured property.

Parameter	Repeatability	Reproducibility
Rate Index, INC	$0.159 - 0.151X$	$0.607 - 0.590X$
Rate Index, DEC	$0.037 - 0.028X$	$0.90 - 0.90X$
VIS100, INC	$-1.38 + 0.12Y$	$0.20 + 0.19Y$
VIS100 DEC	$-1.00 + 0.11Y$	$0.43 + 0.19Y$

Where X is the mean value of rate index, and Y is the mean value of VIS 100 in mPa·s.

VISCOSITY, SAYBOLT UNIVERSAL VISCOSITY:

D 2161

EXPLANATION

This practice covers the conversion tables and equations for converting kinematic viscosity in centistokes (cSt) at any temperature to Saybolt Universal viscosity in Saybolt Universal seconds (SUS) at the same temperature and for converting kinematic viscosity in centistokes at 122 and 210°F to Saybolt Furol viscosity in Saybolt Furol seconds (SFS) at the same temperatures. Kinematic viscosity values are based on water being 1.0038 cSt (mm^3/s) at 68°F(20°C).

A fundamental and preferred method for measuring kinematic viscosity is by use of kinematic viscometers as outlined in Test Method D 445, Test Method for Determination of Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity). It is recommended that kinematic viscosity be reported in centistokes, instead of Saybolt Universal Seconds (SUS) or Saybolt Furol Seconds (SFS). Thus this practice is being retained for the purpose of calculation of kinematic viscosities for SUS and SFS data which appear in past literature. One centistoke equals one millimetre squared per second (mm^2/s).

VISCOSITY, SHEAR OF COAL-TAR AND PETROLEUM PITCHES:
D 5018

EXPLANATION

This test method is useful as one element in establishing the uniformity of shipments. Viscosity is valuable for rheological characterization of binder pitches. Binder pitch imparts consistency to carbonaceous mixes and affects their resistance to deformation. Binder pitch viscosity is important in assessing mix consistency and for evaluating the ease of mix extrusion or molding into artifacts. This test method determines the apparent shear viscosity of coal-tar and petroleum based pitches having a Mettler softening point range of approximately 95 to 120°C. This test method is applicable only for rotational viscometers. Strict adherence to details of the procedure are necessary to comply with the theoretical requirements of this test.

TEST SUMMARY

The apparent shear viscosity is measured via a concentric cylinder viscometer. Apparent shear viscosity is the ratio of shear stress to shear rate in a unidirectional simple shear flow field at steady state conditions. The extrapolated value of apparent shear viscosity at zero shear rate is called shear viscosity.

TEST PRECISION

Repeatability:	12 % of mean
Reproducibility:	36 % of mean

There is no bias.

VISCOSITY-TEMPERATURE RELATIONSHIP OF USED AND SOOT CONTAINING ENGINE OILS:
D 7110

EXPLANATION

The low temperature, low shear viscometric behavior of an engine oil, whether new, used, or sooted, determines whether the oil will flow to the sump inlet screen, then to the oil pump, then to the sites in the engine requiring lubrication in sufficient quantity to prevent engine damage immediately or ultimately after cold temperature starting. Two forms of flow problems have been identified: flow-limited and air-binding behavior. The first form of flow restriction, flow limited behavior is associated with the oil's viscosity; the second, air binding behavior, is associated with gelation.

The temperature scanning technique employed in this test method was designed to determine the susceptibility of the engine oil to the flow limited and air binding response to slow cooling conditions by providing continuous information on the rheological condition of the oil over the temperature range of use. In this way, both viscometric and gelation response are obtained in one test. This test also yields information on parameters called gelation index and gelation index temperature. The first parameter is a measure of the maximum rate of torque increase caused by the rheological response of the oil as the oil is slowly cooled. The second parameter is the temperature at which the gelation index occurs.

A shear rate of approximately 0.2 per sec is produced at shear stresses below 200 Pa. Apparent viscosity is measured continuously as the sample is cooled at a rate of 3°C per hour over the range of −5 to −40°C. Applicability of this test method to petroleum products other than engine oils has not been determined.

TEST SUMMARY

Used and sooted engine oils are analyzed using a special rotational viscometer with analog or digital output to a computer program. A specially made glass stator/metal rotor cell is attached to the viscometer and subjected to a programmed temperature change for both calibration and sample analysis. Following calibration of the rotor/stator set, an approximately 20 mL test sample of a lubricating test oil is poured into the stator and preheated for 1.5 to 2.0 h in an oven or water bath. Shortly after completing the preheating step, the room temperature rotor is put into the stator containing the heated oil and coupled to a torque sensing viscometer head using an adapter to automatically center the rotor in the stator during test. A programmable low temperature bath is used to cool the cell at a specified rate of 3°C/h from −5°C to the temperature at which the maximum torque recordable is exceeded when using a speed of 0.3 r/min for the rotor. After the desired information has been collected, the computer program generates the desired viscometric and rheological values from the recorded data.

TEST PRECISION

Parameter	Repeatability	Reproducibility
Critical Temperature	1.1°C	1.4°C
Gelation Index	11 % of mean	27 % of mean
Gelation Index Temperature	0.7°C	2.8°C
Viscosity	8 % of mean	4 % of mean

Since there is no accepted reference material suitable for determining the bias of this test method, no statement on bias is being made.

VOLATILES CONTAMINANTS IN USED ENGINE OILS: D 3607

EXPLANATION

The removal of volatile materials such as gasoline dilution from used engine oils is especially important if the mechanical shear stability or oxidative stability of the oil is being monitored by measuring a kinematic viscosity change in the oil after it has been used in a gasoline engine. This test method describes a standardized procedure for removing these volatile materials from used engine oils prior to further oil analysis. It also provides an estimate of such volatiles in used engine oils.

TEST SUMMARY

A known weight of sample is heated to 90°C under a nitrogen flow for 4.5 h, cooled, and reweighed.

TEST PRECISION

Repeatability:	0.27 mass %
Reproducibility:	1.40 mass %

There is no bias.

VOLATILES MATTER IN GREEN PETROLEUM COKE: D 6374

EXPLANATION

The volatile matter of petroleum coke affects the density of coke particles and can affect artifacts produced from further processing of the coke. The volatile matter can be used in estimating the calorific value of coke. This test method covers the determination of the volatile matter produced by pyrolysis or evolved when petroleum coke is subjected to the specific conditions of the test method. This test method is empirical and requires the entire test procedure to be closely followed to ensure that the results from different laboratories will be comparable. This method is not satisfactory for determining dedusting material content. Samples having a thermal history above 600°C are excluded from this test.

There are two sources of interferences in this test method. Moisture increases the mass loss, and the moisture free sample weight is decreased by the amount of moisture actually present in the test sample. The particle size range of the analysis sample affects the volatile matter. The coarser the sample, the lower the reported volatile matter will be.

TEST SUMMARY

Volatile matter of a moisture free petroleum coke sample is determined by measuring the mass loss of the coke when heated under the exact conditions of this test method.

TEST PRECISION

Repeatability:	$0.01905 \times (X + 2.826)$
Reproducibility:	$0.06662 \times (X + 2.826)$

Where X is the average of two results in weight percent.

Bias is not known.

VOLATILES MATTER IN PETROLEUM COKE: D 4421

EXPLANATION

The volatile matter in petroleum coke affects the density of coke particles and can affect artifacts produced from further processing of the coke. The volatile matter can be used in estimating the calorific value of coke. Samples having a thermal history above 600°C are excluded. The test method is also not satisfactory for dedusting material content. This test method is empirical and requires the entire test procedure to be closely followed to ensure results from different laboratories to be comparable.

TEST SUMMARY

Volatile matter of a moisture free petroleum coke is determined by measuring the mass loss of the coke when heated under the exact conditions of this procedure. The particle size range of the sample affects the volatile matter. The coarser the sample, the lower the volatile matter will be. Particular care must be taken against mechanical defects in the furnace operations.

TEST PRECISION

This test method has a repeatability of $0.08X$ and a reproducibility of $0.21X$ where X is the average of two results in mass %.

Bias for this test method is not applicable.

VOLATILES PITCH VOLATILITY: D 4893

EXPLANATION

This test is useful in evaluating and characterizing pitch physical properties in comparing the consistency of shipments of sources of supply. Since this test method is empirical, strict adherence of the procedure is necessary.

TEST SUMMARY

An aluminum weighing dish with about 15 g of specimen is introduced into the cavity of a metal block heated and maintained at 350°C. After 30 min during which the volatiles are swept away from the surface of the liquid pitch by preheated nitrogen, the sample is taken out and allowed to cool down in the desiccator. The pitch volatility is determined by the sample weight loss and reported as percent weight loss.

TEST PRECISION

Repeatability:	0.2 %
Reproducibility:	0.3 %

VOLATILITY OF LPG: D 1837

EXPLANATION

Volatility, expressed in terms of the 95 % evaporated temperature of the product, is a measure of the amount of least volatile components present in the product. Coupled with a vapor pressure limit, it serves to ensure essentially single-component products in the cases of commercial grades of propane and butane. When the volatility is coupled with a vapor pressure limit which has been related to density, as in the case of the commercial PB-mixtures, the combination serves to assure essentially two component mixtures for such fuels. When coupled with a proper vapor pressure limit, this measurement serves to assure that special-duty propane products will be composed chiefly of propane and propylene, and that propane will be the major constituent. The presence of hydrocarbon compounds less volatile than those of which the LPG is primarily composed is indicated by an increase in the 95 % evaporated temperature. When the type and concentration of higher boiling components is required, chromatographic analysis should be used.

TEST SUMMARY

The sample is refrigerated by means of a cooling coil and collect 100 mL of liquid in a weathering tube. The sample is allowed to evaporate ("weather") at ambient pressure under specified conditions mat approximate a single plate distillation. When 5 mL of liquid test portion remains, the observed temperature is corrected for barometric pressure and thermometer ice point error, and reported as 95 % evaporation temperature.

TEST PRECISION

Repeatability: 6 °C(1.0 °F)
Reproducibility: 1.0 (1.7 °F) for butane and butane-propane mixtures
3 °C(2.3 °F) for propane

This test method has no bias because the volatility is defined only in terms of this test method.

WATER IN PETROLEUM PRODUCTS:

GENERAL

A knowledge of the water content of petroleum products is important in refining, purchase and sale, and transfer of products, and is useful in predicting the quality and performance characteristics of the products.

There are numerous methods for the determination of water in petroleum products; many specific to a particular product line. Analytically the methods can be grouped as by distillation or by Karl Fischer titrations. Often the choice depends on the levels of water present in the sample. Generally, trace amounts of water must be determined by coulometric Karl Fischer methods. The Karl Fischer titration methods have been in use for over 60 years. They are extremely well documented methods with method interferences well characterized. Generally, these methods work well in "clean" matrices; but there are many potential chemical and physical problems with "real" and complex samples. ASTM Interlaboratory Cross Check Program conducted under the auspices of Coordinating Subcommittee D02.92 shows very poor reproducibility for lubricating oil and lube adpack crosschecks. Almost all participating laboratories in these programs show either high or low bias.

In addition to the D02 sponsored Karl Fischer methods, there are several more methods under the jurisdiction of other ASTM committees. However, many of those are also applicable to the analysis of petroleum products. These methods consist of either manual, potentiometric, or coulometric titrations.

Interferences in Karl Fischer titrations could be of three types:

(1) *Physical*—Electrodes fouling; residual moisture in glassware and reagents; contamination of samples; separation of water layer in storage.
(2) *Chemical*—A number of substances and classes of compounds interfere by condensation and redox reactions. Many of these can be eliminated by adding specific reagents prior to titration. The interfering chemicals include: aldehydes, amines, ammonia, ketones, halogens, mercaptans, metallic O/OH compounds, oxidizers, sulfides.
(3) *Other*—The reagents may themselves react with certain species in the sample. Different brands of pyridine-free reagents have been found to give different results with different chemical samples. The discrepancy appears to be more pronounced in aromatic hydrocarbons than in nonaromatic hydrocarbons. Some of these interferences, in addition to be able to be suppressed by addition of certain reagents, can also be overcome by using vaporizer accessory which physically removes water from the heated matrix as water vapor and then titrates it coulometrically.

Table 12 compares the main characteristics of alternative methods. This table includes some of the non-D02 committee test methods also, since they are relevant to the knowledge of this important technique.

Manual	Potentiometric	Coulometric
D 890	D 1348	D 1123, Test Method B
D 1123, Test Method A	D 1364	D 1533, Test Method B
D 1364	D1533, Test Methods A and C	D 3401, Test Method A
D 1568	D 1631	D 4672, Test Method B
D 2072	D 1744	D 4928
E 203	D 3401, Test Method B	E 1064
	D 4017	D 6304
	D 4377	
	D 4672, Test Method A	
	E 203	
	F 1214	

TABLE 12—*Petroleum Products Related to ASTM Karl Fischer Water Test Methods.*

Test Method	Matrix	Scope	Titration	Interferences	*r*	*R*
D 890-87	Liquid Naval Stores	···	Manual Colorimetry $(\mathrm{I_2{-}SO_2{-}Pyr.})$	···	0.005 %	···
D 1123-93	Engine Coolants	···	A-Manual Colorimetric (Hydranal)	···	0.5 mL	5–15 %
			B-Coulometry		3 ppm	10 ppm
D 1348-89	Cellulose	···	Potentiometry	[a]	0.41	···
D 1364-90	Volatile Solvents	0.5–10 %	Manual Colorimetry or Potentiometry $(\mathrm{I_2{-}SO_2{-}Pyr.})$	[a]	0.015 %	0.027 %
D 1533-88	Insulating Liquid	<200 ppm	A-Potentiometry $(\mathrm{I_2{-}SO_2{-}Pyr.})$	[a]	7 ppm	20 ppm
			B-Coulometry	[a]	3 ppm	10 ppm
			C-Potentiometry with buffer	[a]	···	···
D 1568-91	Alkylbenzene-Sulfonates	···	Manual Colorimetry $(\mathrm{I_2{-}SO_2{-}Pyr.})$	···	0.22 %	0.17 %
D 1631-93	Solvents	0.01–20 %	Potentiometry $(\mathrm{I_2{-}SO_2{-}Pyr.})$	[a]	0.0035	0.021
D 1744-92	LPP	50–1000 ppm	Potentiometry $(\mathrm{I_2{-}SO_2{-}Pyr.})$	[a]	11 ppm	···
D 2072-87	Fatty *N* Compounds	···	Manual Colorimetry	···	···	···
D 3401-92	Halogenated Solvents	2–1000 ppm	A-Coulometry B-Potentiometry	[a]	···	···
D 4017-90	Paints	0.5–70 %	Potentiometry $(\mathrm{I_2{-}SO_2{-}Pyr.})$	[a]	4.7 %	15.0 %
D 4377-93a(IP 356)	Crude Oils	0.02–2 %	Potentiometry $(\mathrm{I_2{-}SO_2{-}Pyr.})$	[a]	$0.034X^{0.33}$	$0.1X^{0.33}$
D 4672-95	Polyols	<0.1–1 %	Potentiometry	[a]	···	···
D 4928-89 (IP 386)	Crude oil	0.005>5 %	Coulometry	[a]	$0.04X^{0.66}$	$0.105X^{0.66}$
E 203-92b	Solids, Liquids	<300 ppm	Potentiometry or Manual Colorimetry	[a]	0.013 %	0.028 %
E 1064-92	Organic Liquids	0–2 %	Coulometry	[a]	5.6 %	17.1 %
F 1214-89y	LPP	50–100 ppm	Potentiometry	[a]	···	···
IP BR/94	LPP	30–1000 ppm	Coulometry	[a]	···	···
D 6304	LPP	10–1000 ppm	A-Coulometry	[a]	$0.04X^{0.6}$	$0.5X^{0.7}$
			B-Evaporative Coulometry		$0.09X^{0.7}$	$0.4X^{0.6}$

LLP = Liquid Petroleum Products.

[a] Interferences normally associated with KF titrations such as mercaptans, sulfides, ketones, aldehydes, etc.

_=Underlined methods of D2 origin or interest.

WATER IN CRUDE OILS BY COULOMETRIC KF TITRATION:

D 4928

(Equivalent Test Methods: IP 386 and ISO 10337)

TEST SUMMARY

This test method is applicable to the determination of water in the range 0.02 to 5 mass % in crude oils. Mercaptan and sulfide interfere. At levels of <500 ppm as sulfur, the interference from these compounds is insignificant. The range of this test method can be extended to 0.005 mass %, but the effects of the mercaptan and sulfide interferences at this level have not been determined. However, it can be expected to be significant at these low water levels.

An aliquot of a homogenized crude oil sample is injected into the titration vessel of a Karl Fischer apparatus in which iodine for the reaction is generated coulometrically at the anode. When all the water has been titrated, excess iodine is detected by an electrometric end point detector, and the titration is terminated. Based on the stoichiometry of the reaction, one mole of iodine reacts with one mole of water, thus the quantity of water is proportional to the total integrated current according to Faraday's law.

The precision of this test method is critically dependent on the effectiveness of the homogenization step. Follow the procedure given in Annexe A1 of Test Method D 4928 to check on the efficiency of the mixer used for homogenization. This test method can use samples taken either gravimetrically or volumetrically.

TEST PRECISION

Sample Taken	Repeatability	Reproducibility
Gravimetry	$0.040X^{0.66}$	$0.105X^{0.66}$
Volumetry	$0.056X^{0.66}$	$0.112X^{0.66}$

Where X is the sample mean.

This precision is valid in the water concentration range of 0.005 to 5 mass %, either gravimetrically or volumetrically.

This test method was found not to have a bias.

WATER IN CRUDE OIL BY DISTILLATION:

D 4006

(Equivalent Test Method: IP 358)

TEST SUMMARY

The sample is heated under reflux conditions with a water immiscible solvent (xylene), which co-distills with the water in the sample. Condensed solvent and water are continuously separated in a trap; the water settles in the graduated section of the trap, and the solvent returns to the distillation flask.

TEST PRECISION

Concentration, %	Repeatability	Reproducibility
0.0 to 0.1 %	See Fig. 18	See Fig. 18
>0.1 %	0.08	0.11

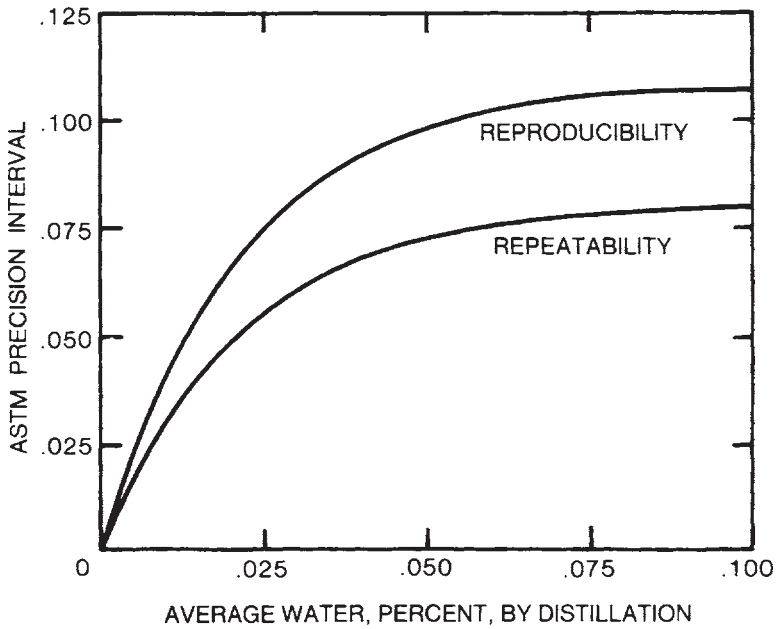

FIG. 21—*Basic Sediment and Water Precision.*

WATER IN CRUDE OILS BY POTENTIOMETRIC KF TITRATION:

D 4377

(Equivalent Test Method: IP 356)

TEST SUMMARY

This test method covers the determination of 0.02 to 2 % in crude oil. Mercaptan and sulfide interfere. At levels <500 ppm as sulfur their interference is insignificant. After homogenizing the crude oil with a mixer, an aliquot in a mixed solvent is titrated to an electrometric end point using either a standard or a pyridine-free Karl Fischer reagent.

TEST PRECISION

KF Reagent	Repeatability	Reproducibility
Standard	$0.034X^{0.33}$	$0.111X^{0.33}$
Pyridine-free	$0.032X^{0.33}$	$0.095X^{0.33}$

Where X is the sample mean from 0.00 to 2 %.

Compared to the results of Test Method D 4006, no significant bias has been found.

FREE WATER, PARTICULATES, AND CONTAMINANTS IN AVIATION FUELS:

D 6986

EXPLANATION

Fuel quality is paramount in aviation fuels because of their critical application. Many successive types of inspections are conducted to ensure quality protection. Rapid, visual inspections carried out at various locations in the fuel supply system are a critical part of the inspection program. Experience has shown that subjective evaluations such as described in this test method form an effective field alert system that is backed by other, more quantitative tests. This test method duplicates much of Test Method D 4176, a test method applicable to all distillate fuels. However, the present test method also includes field methods applicable especially to aviation fuels.

This test method covers two procedures for establishing the presence of free water, solid particulate, and other contaminants in aviation gasoline and aviation turbine fuels. Procedure A uses transparent containers, while Procedure B uses opaque containers. Both procedures are rapid methods for contamination detection, and include ratings of haze appearance and particulate presence. Uncertain or marginal results by either of these procedures would normally result in testing by Test Methods such as D 2276, D 5452, or D 3240 for quantitative determination of contaminants. Particulate determination in appearance tests is sensitive to sampling procedures. The presence of a small number of particles may indicate, for example, that the sample line was not flushed to provide a representative sample. The persistent presence of even a small number of particles, however, may be cause for further investigation depending on the situation.

Experience has shown that an experienced tester using a clear bottle can detect as little as 40 ppm of free, suspended water in the fuel. Thus, a fuel rated as "clear and bright" can still fail lower limits set by quantitative methods. A rater will also have difficulty resolving particles smaller than 40 μm. Smaller particles must be determined by other methods such as D 2276, D 5452, or other chemical tests. Experience has shown the visual appearance of fuel in a white porcelain bucket to be the most suitable method for the detection of dye contaminants or other unusual discoloration. In the U.S., the white porcelain bucket is used to detect the dye.

TEST SUMMARY

Procedure A covers transparent sample containers, including the open jar and the closed circuit sampler, while Procedure B uses opaque containers such as the white bucket. In the open jar procedure, 750 mL of fuel is placed into a clear 1 L container and visually examined. The jar is closed and the sample is swirled and examined for visual sediment and water at the bottom of the vortex. Additionally, fuel clarity may be rated by placing a standard bar chart behind the sample and comparing its visual appearance with the standard haze rating photographs. The presence or absence of free water and of particulates is reported.

In the closed circuit sample procedure, approximately 3500 mL of fuel is placed into the sampler and is examined for clarity and for visual sediment or water droplets on the bottom of the sampler. Additionally, fuel clarity may be rated by placing a standard bar chart behind the sample and comparing its visual appearance with the standard haze rating photographs. The presence or absence of free water and of particulates is reported.

In the white bucket procedure, fuel to a depth of approximately 15 cm is collected in a white porcelain coated or stainless steel bucket. The sample is examined for solids or sediment, or both, on the bottom of the bucket. Sample clarity can be checked by the appearance of a small, shiny coil on the bucket's bottom. If fuel is dry, the raised letters on the coil should be easily readable. The amount of sediment can be described by a letter category using a rating guide. In both procedures, the sample is inspected for color or other unusual appearance. Field inspection procedures are performed immediately after sampling at fuel handling temperature conditions.

TEST PRECISION

No precision to these procedures can be determined since they are not quantitative procedures. Nor can a justifiable statement can be made regarding the bias of this method because a fuel haze can be the result of a number of causes and a relationship with any single absolute quantitative measurement is not possible.

WATER FREE WATER IN DISTILLATE FUELS: D 4176

TEST SUMMARY

Fuel specifications often contain the requirement that it be *clear and bright and free of visible particulate matter*. This test method provides two procedures to cover that requirement. This test method is applicable to distillate fuels having distillation end points below 400°C and an ASTM color of 5 or less. Both procedures are performed immediately after sampling and at storage temperature conditions.

PROCEDURE A—is a rapid pass/fail method for contamination. Approximately 900 mL of fuel is placed into a 1–L jar of clear glass, and is examined visually for clarity. The sample is then swirled and examined for visual sediment or water drops below the vortex.

PROCEDURE B—provides a gross numerical rating of haze appearance. Approximately 900 mL of fuel is placed into a 1–L jar of clear glass and is examined visually for clarity. Fuel clarity is rated by comparing against a standard bar chart and haze rating photos. The sample is then swirled and examined for visual sediment or water drops below the vortex.

TEST PRECISION

Procedure A is a pass/fail test and precision statement is not appropriate.

Procedure B has a repeatability of 1 number and a reproducibility of 2 numbers rating.

No bias statement is possible for this test.

WATER FREE WATER IN MID-DISTILLATE FUELS: D 4860

TEST SUMMARY

This test is similar to D 4176 just described. The color of sample does not affect the measurement. The test uses a rapid, portable means of visually inspecting for particulate matter and rating numerically for free water in aviation turbine and distillate fuels, both in field and in a laboratory.

The inspection for particulate matter is done the same way as in Test Method D 4176. A numerical rating for free water is obtained by filtering a portion of the fuel sample at a programmed rate through a standard fiberglass coalescer/filter. A portion of the effluent is used as a reference against an unfiltered portion to obtain the rating.

When a fuel is visually inspected at or below the cloud point temperature of the fuel, small amounts of solid wax particles can be confused with a water-induced haze or cloudiness. The presence of free water or particulate can be obscured and missed during visual inspection of the fuel, if the ASTM color rating is greater than five.

TEST PRECISION

Visual particulate matter is a pass-fail test, and a statement of precision is not appropriate. The repeatability for the numerical rating of free water is 6, and a reproducibility is 7.

This test method has no bias.

WATER IN PETROLEUM PRODUCTS AND LUBRICANTS BY COULOMETRIC KARL FISCHER TITRATION: D 6304

TEST SUMMARY

This test method covers the direct determination of water in the range of 10 to 25 000 mg/kg entrained water in petroleum products and hydrocarbons using automated instrument. This test method is applicable to additives, lube oils, base oils, automatic transmission fluids, hydrocarbon solvents, and other petroleum products. By proper choice of the sample size, this test method may be used for the determination of water from mg/kg to percent level concentrations. The normal chemical interferences in Karl Fischer titrations also apply to this test method.

The sample injection in the titration vessel can be done volumetrically or gravimetrically. The instrument automatically titrates the sample and displays the result at the end of the titration. Viscous samples can be analyzed by using a water vaporizer accessory that heats the sample in the evaporation chamber, and the vaporized water is carried into the Karl Fischer titration cell by a dry inert carrier gas.

TEST PRECISION

Injection	Volumetric	Gravimetric
Repeatability	$0.8852X^{0.7}$	$0.03813X^{0.6}$
Reproducibility	$0.5248X^{0.7}$	$0.4243X^{0.6}$

Where X is the mean of duplicate measurements. The values are in volume or mass percent.

This test method has no bias.

WATER IN PETROLEUM PRODUCTS BY DISTILLATION METHOD:
D 95
(Equivalent Test Methods: IP 74, ISO 3733, DIN 51582, JIS K 2275, and AFNOR T60-113)

TEST SUMMARY

The sample is heated under reflux with a water-immiscible solvent, which co-distills with the water in the sample. Condensed solvent and water are continuously separated in a trap, the water settling in the graduated section of the trap, and the solvent returning to the still.

TEST PRECISION

	Collected Water, mL	Difference, mL
Repeatability	0.0–1.0	0.1
	1.1–25	0.1 or 2 % of Mean, whichever is greater.
Reproducibility	0.0–1.0	0.2
	1.1–25	0.2 or 10 % of Mean, whichever is greater.

WATER BY KARL FISCHER REAGENT:
D 1744

TEST SUMMARY

This test method covers the determination of water in the concentration from 50 to 1000 ppm in liquid petroleum products. The material to be analyzed is titrated with standard Karl Fischer reagent to an electrometric end point. Free alkali, oxidizing and reducing agents, mercaptans, certain basic nitrogenous substances, or other materials that react with iodine, interfere. One part per million of sulfur as mercaptan causes an error in the titration, equivalent to approximately 0.2 ppm of water.

TEST PRECISION

Repeatability:	50 to 1000 ppm water content =11 ppm
Reproducibility:	Not determined

WATER REACTION OF AVIATION FUELS:
D 1094
(Equivalent Test Methods: IP 289, ISO 6250, DIN 51415, and AFNOR M07-050)

TEST SUMMARY

When applied to aviation gasoline, water reaction volume change reveals the presence of water-soluble components such as alcohols. When applied to aviation turbine fuels, water reaction interface rating reveals the presence of relatively large quantities of partially soluble contaminants such as surfactants.

A sample of the fuel is shaken, using a standardized technique, at room temperature with a phosphate buffer solution in scrupulously cleaned glassware. The cleanliness of the glass cylinder is tested. The change in the volume of the aqueous layer and the appearance of the interface are taken as the water reaction of the fuel.

TEST PRECISION

This is a qualitative test, and statements of precision or bias are not appropriate.

WATER RESISTANCE OF LUBRICATING GREASE: D 4049

TEST SUMMARY

This test method is used to evaluate the ability of a grease to adhere to a metal surface when subjected to direct water spray. The results of this test method correlate with the operations involving direct water spray impingement such as steel mill roll neck bearing service. The grease is coated on a stainless steel panel and sprayed with water at 100°F at a pressure of 276 kPa for 5 min. The amount of grease remaining on the panel after the test is a measure of the resistance of grease to water spray.

TEST PRECISION

Repeatability:	6 %
Reproducibility:	18 %

There is no bias.

WATER AND SEDIMENT IN FUEL OILS: D 1796

(Equivalent Test Methods: IP 75, ISO 3734, DIN 51793, and AFNOR M07-020)

TEST SUMMARY

This test method is valid in the range from 0 to 30 % by volume. Equal volumes of fuel oil and water saturated toluene are placed in each of the two cone shaped centrifuges. After centrifugation for 10 min at a rate to give between 500 and 800 relative centrifugal force (rcf), the volume of the higher gravity water and sediment layer at the bottom of the tube is read.

TEST PRECISION

See the test precision in Fig. 22.

The bias of this test method has not been determined.

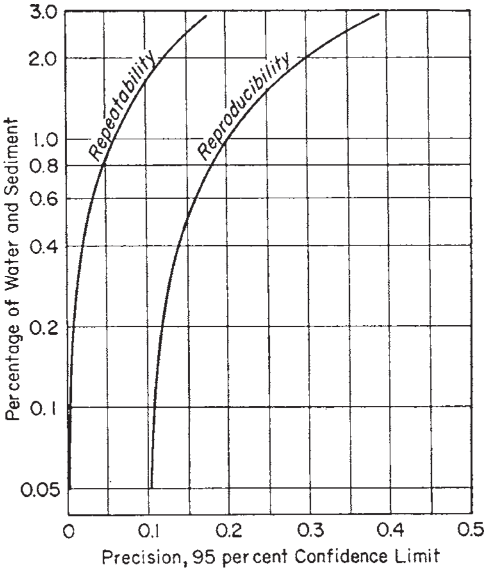

FIG. 22—*Precision Curves for Centrifuge Tube Methods.*

WATER AND SEDIMENT IN CRUDE OIL:
D 4007
(Equivalent Test Method: IP 359)

TEST SUMMARY

See the details for Test Method D 1796.

TEST PRECISION

See the test precision in Fig. 23 at the concentration level of 0.0 to 0.3 % water. From 0.3 to 1.0 % water, the repeatability is 0.12, and the reproducibility 0.28.

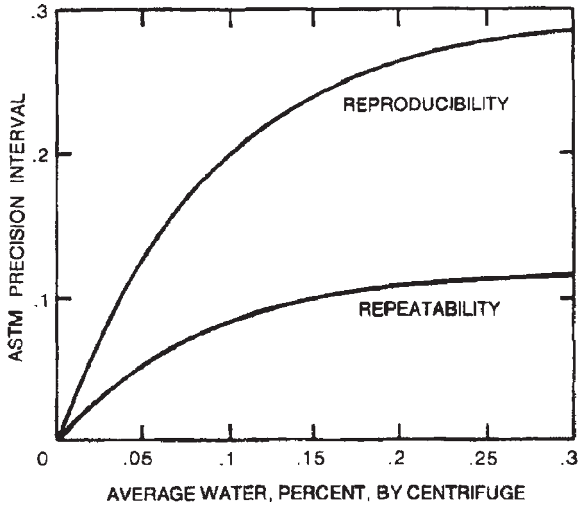

FIG. 23—*Basic Sediment and Water Precision.*

WATER SEPARATION CHARACTERISTICS OF DIESEL FUELS BY PORTABLE SEPARATOMETER:
D 7261

EXPLANATION

This test method provides a measure of the presence of surfactants in diesel fuels, and can be performed in the field or in a laboratory. It is a rapid portable means to rate the diesel fuels to release entrained or emulsified water when passed through fiberglass coalescing material. Similar to Test Method D 3948 used for jet fuels, this test method can detect traces of some refinery treating chemicals left in the fuel. It can also detect surface active substances added to or picked up by the fuel during handling from point of production to point of use.

Certain additives, which can act as weak surfactants, give a slightly reduced DSEP rating. Other substances which are strong surfactants give much lower DSEP ratings. Results from this test method do not have a known relationship to the rate of water settling in the tanks. The Micro-Separator has a measurement range from 50 to 100. Values obtained outside these limits are undefined and invalid. This test method is applicable to diesel fuels such as D 975 Grade No. 1 and Grade No. 2 of all sulfur levels, and MIL-F-16884, naval distillate fuel (NATO F-76).

TEST SUMMARY

A 50 mL water/fuel sample emulsion is created in a syringe using a high-speed mixer. The emulsion is then expelled from the syringe at a programmed rate through a standard fiberglass coalescer and the effluent is analyzed for uncoalesced water by a light transmission measurement. The results are reported on a 0 to 100 scale to the nearest whole number; however, the effective range of the test equipment is from 50 to 100. Higher ratings indicate that water is easily coalesced, implying that the fuel is relatively free of surfactants.

Interferences—Any suspended particles, whether solids or water droplets or haze, in a sample will interfere with this test method. Non-hydrocarbon components such as oxygenates, especially alcohols, or emulsified water have not been verified for this test method and will likely interfere.

TEST PRECISION

The repeatability and reproducibility of this test method has not been obtained at this time. The procedure in this test method has no bias because the value of DSEP is defined only in terms of this test method.

WATER SEPARATION CHARACTERISTICS OF AVIATION TURBINE FUELS:
D 3948

EXPLANATION

This test method provides a rapid, portable means to measure the presence of surfactants in aviation turbine fuels using a portable separatometer. The instrument has a measurement range of 50 to 100. There are two modes of operation of the instrument. The primary difference between them is the rate of fuel flow through the fiberglass coalescing material. The lapsed time required to force the emulsion through the coalescer cell is 45 s for Mode A and 25 s for Mode B. Selection of Mode A or B depends upon the specific fuel and specification requirements.

TEST SUMMARY

A water-fuel sample emulsion is created in a syringe using a high-speed mixer. The emulsion is then expelled from the syringe at a programmed rate through a standard fiber-glass coalescer and the effluent is analyzed for un-coalesced water by a light transmission measurement. The results are reported on a 0 to 100 scale to the nearest whole number. High numbers indicate the water is easily coalesced, implying that the fuel is relatively free of surfactants. A test can be performed in 5 to 10 min .

TEST PRECISION

See the precision of different fuel types in Figs. 24 and 25.

This test method has no bias.

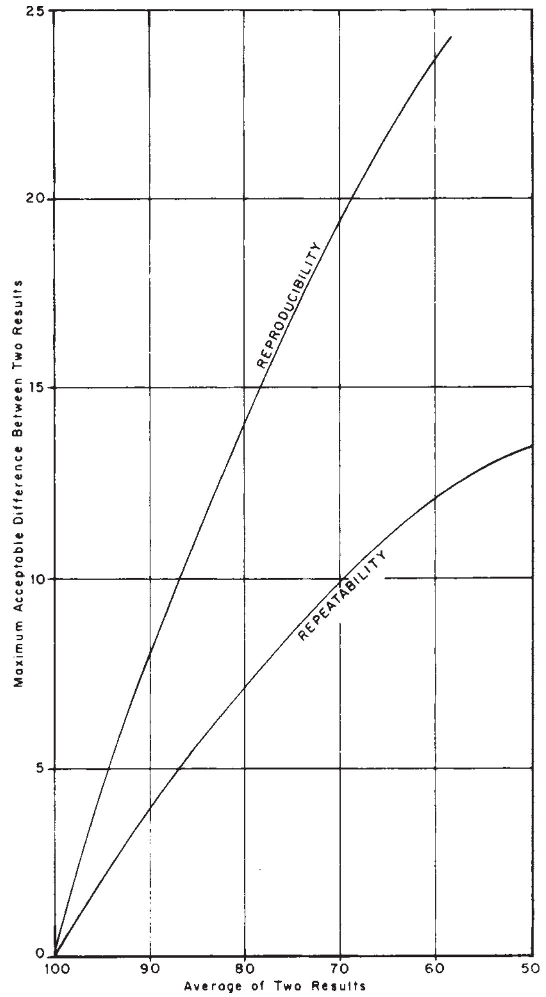

FIG. 24—*Reference Fuels—MSEP-A (Mode A Operation) Variation of Repeatability and Reproducibility of MSEP-A Ratings Obtained for Reference Fuels (Jet A, Jet A-1, MIL JP 5, MIL JP 7, and MIL JP 8) Containing a Dispersing Agent.*

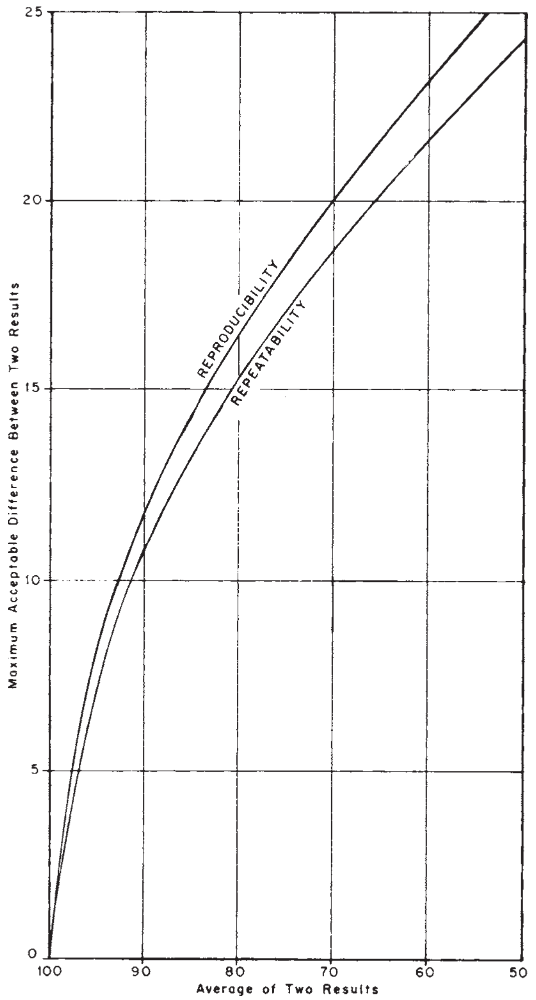

FIG. 25—*Field Samples—MSEP-A (Mode A Operation) Variation of Repeatability and Reproducibility of MSEP-A Ratings Obtained for Field Samples (Jet A, Jet A-1, MIL JP 5, MIL JP 7, and MIL JP 8).*

WATER SEPARABILITY OF PETROLEUM OILS: **D 1401** (Equivalent Test Methods: IP 412, ISO 6614, and AFNOR T60-125)

EXPLANATION

This test method measures the ability of petroleum oils or synthetic fluids to separate from water. It is used for specification of new oils and monitoring of in-service oils.

TEST SUMMARY

A 40–mL sample and 40 mL of water are stirred for 5 min at 54°C in a graduated cylinder. The time required for the separation of the emulsion thus formed is recorded. If complete separation or emulsion reduction to 3 mL or less does not occur after standing for 30 min, the volumes of oil (or fluid), water, and emulsion remaining at the time are reported.

TEST PRECISION

See Fig. 26 for precision.

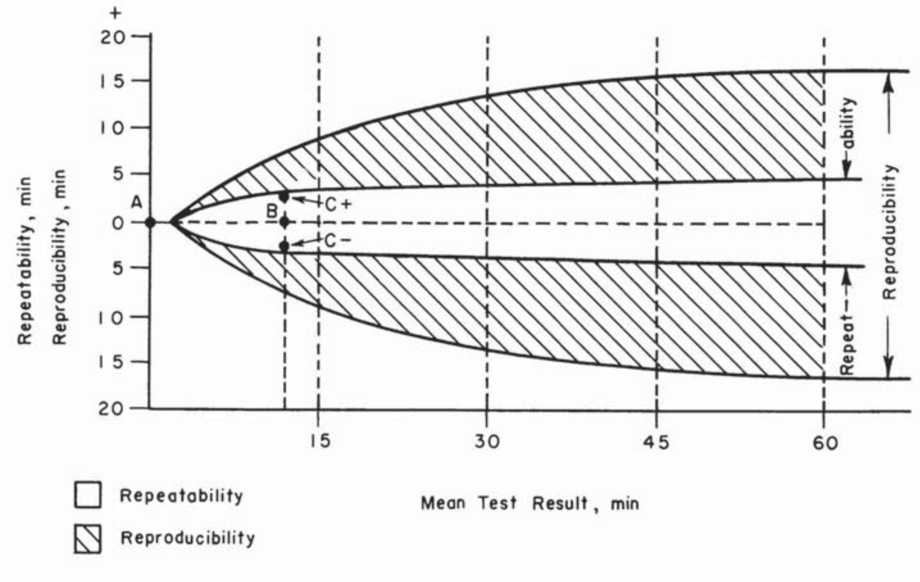

FIG. 26—*Chart for Determining Test Precision.*

WATER SEPARATION CHARACTERISTICS OF KEROSENE-TYPE AVIATION TURBINE FUELS CONTAINING ADDITIVES BY PORTABLE SEPAROMETER: D 7224

EXPLANATION

This test method covers a rapid portable means for field and laboratory use to rate the ability of kerosene-type aviation turbine fuels, both neat and those containing additives, to release entrained or emulsified water when passed through fiberglass coalescing material. This test method is applicable to kerosene-type aviation turbine fuels including jet A and jet A-1 as described in Specification D 1655, JP-5, JP-7, JP-8, and JP-8 + 100. This test method yields approximately the same low MSEP rating as Test Method D 3948 for fuels that contain strong surfactants as well as the products mentioned above. The Micro-Separometer has an effective measurement range from 50 to 100. Values obtained outside this limit are undefined and invalid.

TEST SUMMARY

A water-fuel sample emulsion is created in a syringe using a high-speed mixer. The emulsion is then expelled from the syringe at a programmed rate through a specific fiberglass coalescer, the MCell Coalescer, and the effluent is analyzed for uncoalsced water (i.e., dispersed water droplets) by a light transmission measurement. A test can be performed in 5 to 10 min.

INTERFERENCES—Any suspended particles, whether solid or water droplets or haze, in a fuel sample will interfere with this test method, which utilizes light transmission of a fuel sample after emulsification with water and subsequent coalescence.

TEST PRECISION

See figure to the right for the repeatability and reproducibility obtained with this test method. This procedure in this test method has no bias because the value of MSEP is defined only in terms of this test method.

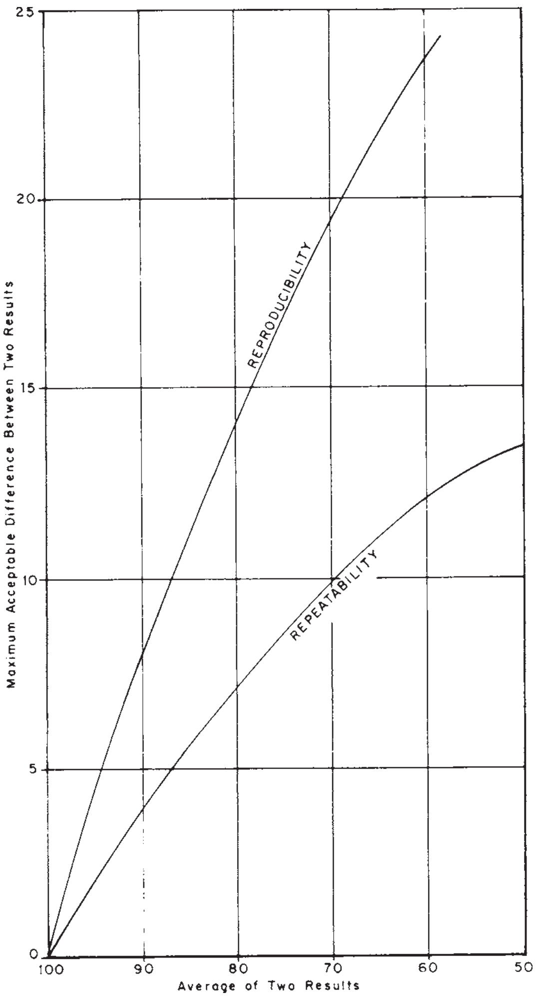

FIG. 27—*Reference Fuels—Variation of Repeatability and Reproducibility of MSEP Ratings Obtained for Reference Fuels (Jet A, Jet A-1, JP-5, JP-7, and JP-8) Containing a Dispersing Agent.*

WATER SOLUBILITY IN HYDROCARBONS AND ALIPHATIC ESTER LUBRICANTS: D 4056

EXPLANATION

This test method covers a procedure for estimating the equilibrium solubility of water and its vapor in hydrocarbon and aliphatic ester lubricants, at temperatures between 277 and 373 K. This test method is limited to liquids of low to moderate polarity and hydrogen bonding, with predicted solubilities not over 1000 ppm by weight in hydrocarbons, or 30 000 ppm by weight in oxygenated compounds, at 298 K. Olefins, nitriles, nitro compounds, and alcohols are specifically excluded. This test method is recommended only for liquids not containing widely different chemical species; for example, blends of esters with hydrocarbons, and lubricants containing detergents, dispersants, rust preventives, or load carrying additives are excluded.

TEST SUMMARY

The solubility is calculated from data for density, refractive index, and molecular weight of a hydrocarbon. For an ester, saponification number is also required.

TEST PRECISION

The precision of this test method is entirely dependent on that of the test methods used to measure the four components that go into the calculations.

WATER IN SOLVENTS BY KARL FISCHER TITRATION: D 1364

EXPLANATION

This test method covers the determination of water in any proportion in volatile solvents and chemical intermediates used in paint, varnish, lacquer, and related products.

This test method is not applicable in the presence of mercaptans, peroxides, or appreciable quantities of aldehydes or amines.

This test method is based essentially upon the reduction of iodine by sulfur dioxide in the presence of water. This reaction can be used quantitatively only when pyridine and an alcohol are present to react with the sulfur trioxide and hydriodic acid produced according to the following reactions:

$$H_2O + I_2 + SO_2 + 3C_5H_5N \rightarrow 2C_5H_5N \cdot HI + C_5H_5 \cdot SO_3$$

$$C_5H_5N \cdot SO_3 + ROH \rightarrow C_5H_5N \cdot HSO_4R$$

TEST SUMMARY

To determine water, Fischer reagent (a solution of iodine, pyridine, and sulfur dioxide, in the molar ratio of 1+10+3) dissolved in anhydrous 2-methoxyethanol is added to a solution of the sample in anhydrous pyridine-ethylene glycol (1+4) until all water present has been consumed. This is evidenced by the persistence of the orange-red end-point color; or alternatively by an indication on a galvanometer or similar current-indicating device which records the depolarization of a pair of noble-metal electrodes. The reagent is standardized by titration of water.

TEST PRECISION

Repeatability:	0.015 % absolute
Reproducibility:	0.027 % absolute

WATER TOLERANCE OF GASOLINE-ALCOHOL BLENDS: D 6422

EXPLANATION

Gasoline-alcohol blends have a very limited ability to retain water in solution or in a stable suspension, and if the amount of water in the blend exceeds this limit, the fuel will separate into a lower aqueous and an upper hydrocarbon phase. Temperature is the critical factor governing the ability of a fuel to retain water without separating. This test method determines the maximum temperature at which the fuel will separate. This test method is applicable to gasoline-alcohol blends for use as fuels in spark-ignition engines that contain saturated C_1 to C_4 alcohols only. This test method does not apply to fuels that contain an alcohol as the primary component, such as M85 or E85, or to gasoline-ether blends.

TEST SUMMARY

A fuel sample is cooled at a controlled rate to its lowest expected storage or use temperature and is periodically observed for phase separation. The apparatus of Test Method D 2500 or a dry ice-isopropyl alcohol bath may be used. A maximum cooling rate of $2°C/min$ is specified because phase separation in gasoline-alcohol blends can have a relatively long but unpredictable induction period.

TEST PRECISION

The precision and bias of this test method are currently being determined.

WATER UNDISSOLVED IN AVIATION TURBINE FUELS: D 3240

EXPLANATION

Undissolved (free) water in aviation fuel can encourage the growth of microorganisms and subsequent corrosion in the tanks of aircraft and can also lead to icing of filters in the fuel system. The usual range of the test reading covers from 1 to 60 ppm of free water. This test method does not detect dissolved water, and thus test results for comparable fuel streams can vary with fuel temperature and the degree of water solubility in the fuel.

TEST SUMMARY

A measured sample of fuel is passed through a uranine dye-treated filter pad. Undissolved water in the fuel will react with the uranine dye. When the pad is subsequently illuminated by ultraviolet light, the dye previously contacted by water will fluoresce a bright yellow with the brightness increasing for increasing amounts of free water in the fuel. The UV light-illuminated pad is compared to a known standard using a photocell comparator, and the free water in the fuel is read out in ppm by volume. By varying the sample size, the range of the test method can be increased.

TEST PRECISION

Repeatability:	$0.32X$
Reproducibility:	$0.47X$

Where X is the average of two results in ppm by volume over the range from 1 through 60 ppm/v.

Bias has not been determined.

WATER WASHOUT CHARACTERISTICS OF LUBRICATING GREASES:
D 1264
(Equivalent Test Methods: IP 215, ISO 11009, and DIN 51807 T2)

EXPLANATION

This test method covers the evaluation of the resistance of a lubricating grease to washout by water from a bearing, when tested at 38 and 79°C (100 and 175°F) under the prescribed laboratory conditions. No correlation with field service has been established.

TEST SUMMARY

The grease is packed in a ball bearing that is then inserted in a housing with specified clearances, and rotated at 600±30 r/min. Water, controlled at the specified test temperature, impinges on the bearing housing at a rate of 5±0.5 mL/s. The amount of grease washed out in 1 h is a measure of the resistance of the grease to water washout.

TEST PRECISION

Temperature, °C	Repeatability	Reproducibility
38	0.8 $(X+2)$	1.4 $(X+2)$
79	0.6 $(X+4.6)$	1.1 $(X+4.6)$

Where X is the average of two results in percent.

This test method has no bias.

WAX APPEARANCE POINT OF DISTILLATE FUELS:
D 3117

EXPLANATION

Wax appearance point is the temperature at which wax crystals begin to precipitate out of a fuel under specified cooling conditions. The presence of wax crystals in the fuel may restrict flow or plug the fuel filter. In critical fuel systems, wax appearance point may define the lower limits of acceptable operability. This test method is applicable to burner fuels, diesel fuels, and turbine engine fuels in the range from −26 to +2°C. It is applicable to dark colored oil if the stirrer can be seen under the illumination.

TEST SUMMARY

A specimen of distillate fuel is cooled under prescribed conditions while being stirred. The temperature at which wax first appears is considered the wax appearance point.

TEST PRECISION

Repeatability:	0.8°C
Reproducibility:	2.2°C

Bias has not been determined.

WAX APPLIED DURING CURTAIN COATING OPERATION:
D 3708

EXPLANATION

Wax coatings are applied to corrugated board to provide a barrier against moisture or other penetrates or to provide improved appearance or abrasion resistance. These functional properties are influenced by the amount of wax present on the surface. During curtain coating operations, major portions of the wax will congeal on the surface, while a minor portion will penetrate and become embedded in the fibers of the facing. This method measures the total weight of wax applied to the board. The amount actually remaining on the surface of the corrugated board can be determined by Test

Method D 3521. The uniformity of wax application across the width of the curtain coater may also be determined using this technique by passing test combinations (blanks) under the curtain at various locations, that is, left side, right side.

Related method for determining the weight of wax coating include Test Method D 3521, D 3522, and D 3344.

TEST SUMMARY

A folded sheet of paper is attached to production corrugated board, the combination is run through the curtain coater, and subsequently the applied weight of the wax on the sheet of paper is determined.

TEST PRECISION

Repeatability is 10 % of the mean. No reproducibility data are available. This procedure has no bias because the value of weight of wax applied can be defined only in terms of this test method.

WAX CONTENT OF CORRUGATED PAPERBOARD: D 3344

EXPLANATION

Many of the functional properties of wax-treated corrugated paperboard and cartons are dependent on the amount of wax present. In the case of the wax-saturated or wax-impregnated paperboard, the principle concern is with the weight of wax used relative to the weight of the paperboard present, that is, the weight percent content or pickup. In some applications the saturating wax may be deposited in the three elements of the corrugated board in such a way as to individually control the amount in each element, that is, the medium and the two facings.

In the case of the wax-coated corrugated paperboard, the principle concern is the weight of the wax on the board surface per unit area. The functional values of the wax coatings as a barrier or a decorative coating are dependent, in part, on the amount of wax in the continuous surface layer, relative to the area covered. The weight of the coating relative to the weight of substrate is not usually a concern with regard to product quality. This test method is applicable to specimens that have been waxed by either impregnation (saturation) operations, or combinations of such operations.

TEST SUMMARY

The wax from the board is extracted twice with 1,1,1-trichloroethane, and the extract evaporated to dryness to determine the total quantity of wax associated with the corrugated board specimen. Use of a Soxhlet extraction technique may improve precision of the results.

TEST PRECISION

Repeatability:	5 % of the mean
Reproducibility:	12 % of the mean

This procedure has no bias because the total wax content of corrugated paper and paperboard can be defined only in terms of this test method.

WEAR CHARACTERISTICS OF PETROLEUM HYDRAULIC FLUIDS:
D 6973

EXPLANATION

This test method is an indicator of the wear characteristics of petroleum hydraulic fluids operating in a constant volume vane pump. Excessive wear in vane pumps could lead to malfunction of hydraulic systems in critical industrial or mobile hydraulic applications.

TEST SUMMARY

Hydraulic fluid in the amount of 190±4 L is circulated through a rotary vane pump system for 50 h at a pump speed of 2400±20 r/min, and a pump outlet pressure of 20.7±0.2 MPa. Fluid temperature at the pump inlet is 95±3°C. An ISO Grade 32 or 10W viscosity is required. The cam ring and all ten vanes are individually weighed before and after the test. The weight loss of the cam ring is reported with the combined weight loss of all ten vanes. The intra-vanes (inserts) are not part of the required weight loss measurements and should be separately measured, if desired. Other reported values are fluid cleanliness before and after the test, and initial and final flow rates. Prior to installing the hydraulic test fluid into the rig, a stand flush is required to remove any contaminants. A minimum of quantity of 190±4 L of fluid made of the same chemical formulation as the test fluid, is required for the stand flush. Therefore the total quantity of oil required for the test is 380 L.

TEST PRECISION

Work is underway to determine the repeatability, reproducibility, and bias of this test method.

WEAR CHARACTERISTICS OF NON-PETROLEUM AND PETROLEUM HYDRAULIC FLUIDS:

D 7043

EXPLANATION

This test method is an indicator of the wear characteristics of petroleum and non-petroleum hydraulic fluids operating in a constant volume vane pump. Excessive wear in vane pumps could lead to malfunction of hydraulic systems in critical applications. This test method covers a constant volume vane pump test procedure operated at 1200 r/min and 13.8 MPa.

TEST SUMMARY

An amount of 18.9±0.5 L of a hydraulic fluid is circulated through a rotary vane pump system for 100 h at a pump speed of 1200±60 r/min, and a pump outlet pressure of 13.8±0.3 MPa. Fluid temperature at the pump inlet is 66±3°C for all water glycols, emulsions, and other water containing fluids and for petroleum and synthetic fluids of ISO Grade 46 or lighter. A temperature of 80±3°C is used for all synthetic and petroleum fluids. The total quantity of test oil required for a run is 26.5 L. The result obtained is the total mass loss from the cam ring and the twelve vanes during the test. Other reported values are initial flow rate and final flow rate.

TEST PRECISION

The precision and bias of this test method is being determined. However, from a preliminary statistical examination of the round robin data collected it appears that the test repeatability is 75 mg.

WEAR CHARACTERISTICS OF LUBRICATING FLUID (FOUR BALL METHOD):

D 4172

EXPLANATION

This test method covers a procedure for making a preliminary evaluation of the anti-wear properties of fluid lubricants in sliding contact by means of the Four-Ball Wear Test Machine. No attempt has been made to correlate this test with balls in rolling contact or in field performance. This instrument is available from Falex Corporation. The evaluation of lubricating grease using the same machine is described in Test Method D 2266.

TEST SUMMARY

Three steel balls are clamped together and covered with the test lubricant. A fourth ball is pressed with a force of 147 or 392 N into the cavity formed by the three clamped balls for three-point contact. The lubricant temperature is maintained at 75°C, and the fourth ball is rotated at 1200 r/min for 60 min. Lubricants are compared by using the average size of the scar diameters worn on the three lowered clamped balls.

TEST PRECISION

Repeatability:	0.12 mm scar diameter difference
Reproducibility:	0.28

There is no bias.

WEAR CHARACTERISTICS PREVENTING PROPERTIES OF LUBRICATING GREASES:

D 3704

EXPLANATION

This test method is used to differentiate between greases having high, medium, and low wear preventive properties using Falex block-on-the ring friction and wear test machine.

TEST SUMMARY

The tester is operated with a steel test ring oscillating against a steel test block. Test speed, load, angle of oscillation, time and specimen surface finish and hardness can be varied to simulate field conditions. The width of the wear scar, developed on the test block from contact with the oscillating test ring, is measured.

TEST PRECISION

Repeatability:	23 % of the mean
Reproducibility:	39 %

There is no bias.

WEAR CHARACTERISTICS OF LUBRICATING GREASE (FOUR BALL METHOD):

D 2266

(Equivalent Test Methods: IP 239, ISO 11008, and DIN 51350)

EXPLANATION

The four ball wear test method can be used to determine the relative wear preventing properties of greases in sliding steel-on-steel applications under specific test conditions. If the test conditions are changed, the relative ratings may be different. The test is not intended to predict wear characteristics with metal combinations other than steel-on-steel. This test method cannot be used to differentiate between extreme pressure and nonextreme pressure greases. No correlation has been established between this test and field service.

TEST SUMMARY

A steel ball is rotated under load against three stationary steel balls having grease lubricated surfaces. The diameters of the wear scars on the stationary balls are measured after completion of the test.

TEST PRECISION

Repeatability:	0.20 mm
Reproducibility:	0.37 mm

There is no bias.

WEAR CHARACTERISTICS OF HYDRAULIC FLUIDS:
D 2882

EXPLANATION

This test method is an indicator of the wear characteristic of petroleum and nonpetroleum hydraulic fluids operating in a constant volume vane pump. Excessive wear in vane pumps could lead to malfunction of hydraulic systems in critical applications.

TEST SUMMARY

Three gallons of a hydraulic fluid (the test requires a 5-gal sample of oil for the total run) is circulated through a rotary vane pump system for 100 h at a pump speed of 1200 r/min, and a pump outlet pressure of 13.79 MPa. Fluid temperature at the pump inlet is 65.6°C for all water glycols, emulsions, and other water containing fluids and for petroleum and synthetic fluids with viscosities of 46 cSt or less at 40°C. A temperature of 79.4°C is used for all other synthetic and petroleum fluids. The result obtained is the total cam ring and vane weight loss during the test.

TEST PRECISION

The precision and bias of this test method are being developed. In the interim, use Fig. 28.

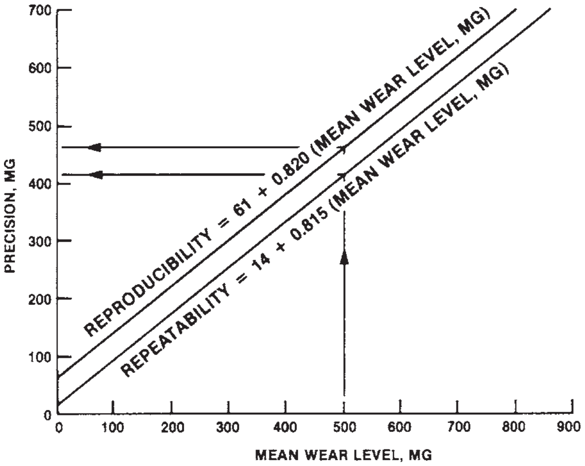

FIG. 28—*Test Precision Relationships.*

WEAR LIFE OF SOLID FILM LUBRICANTS:
D 2981

EXPLANATION

This test method is used for determining the wear life properties of bonded solid film lubricants under oscillating motion by means of a block-on-ring friction and wear testing machine. This test method differentiates between bonded solid lubricants with respect to their wear life. If the test conditions are changed, relative wear life may change and relative ratings of the bonded solid film lubricants may be different.

TEST SUMMARY

The machine is operated using a coated steel testing ring oscillating against a steel test block. The oscillating speed is 87.5 cpm at a 90° arc. The specimens are worn-in for 1 min at 30 lb normal load. Wear-in is followed by application of a normal load of 630 lb for the duration of the test. One measurement is made. Wear life is defined as the number of cycles required for the frictional force to rise to a predetermined value.

TEST PRECISION

Repeatability:	25 % of mean of wear life
Reproducibility:	33 %

Bias has not been determined.

WEAR CHARACTERISTICS OF TRACTOR HYDRAULIC FLUIDS:
D 4998

EXPLANATION

Many modern tractor designs use hydraulic fluid to lubricate the transmission and final drive gears. This test method is used to screen the suitability of the tractor hydraulic fluids for gear wear. Although primarily applicable for tractor hydraulic fluids, it may be suitable for other applications.

TEST SUMMARY

A modified FZG gear oil test machine is operated for 24 h under controlled conditions of speed (100 r/min), load (tenth stage), and temperature (121°C). Test gears are lubricated with the test oil. The test gears are weighed and visually examined before and after the test. The gear weight loss and the visually observed damage to the gear teeth are used to evaluate the wear obtained with the test fluid.

TEST PRECISION

Repeatability:	27.4 mg
Reproducibility:	43.2 mg

There is no bias.