



Standard Guide for Characterizing Hydrocarbon Lubricant Base Oils¹

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1. Scope*

1.1 This guide suggests physical, chemical, and toxicological test methods for characterizing hydrocarbon lubricant base oils derived from various refining processes including re-refining used oils and refining crude oil. This guide does not contain limits nor does it purport to cover all tests which could be employed; rather, it represents the first step in better describing important parameters of lubricant base oils affecting lubricant performance and safe handling. Tests have been identified to characterize the composition and performance of base oils in addition to verifying their consistency. Undesirable components have also been identified with a range of typical levels. These are not limits. It is the responsibility of the buyer and seller to determine and agree upon the implementation of this guide.

1.2 This guide applies only to base oils and not to finished lubricants.

1.3 Base oils containing detectable levels of esters, animal fats, vegetable oils, or other materials used as, or blended into, lubricants are not covered by this guide.

1.4 This guide is relevant to base oils composed of hydrocarbons and intended for use in formulating products including automotive and industrial lubricants. Although not intended to cover all base oil viscosity grades, this guide does cover the majority of viscosities that would be used in both automotive and industrial oil formulations. These base oils would typically have a viscosity of approximately 2 mm²/s to 40 mm²/s (cSt) at 100 °C (50 SUS to 3740 SUS at 100 °F).

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

¹ This guide is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.P0 on Recycled Products.

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2. Referenced Documents

2.1 ASTM Standards:²

- D91 Test Method for Precipitation Number of Lubricating Oils
- D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester
- D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
- D97 Test Method for Pour Point of Petroleum Products
- D130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test
- D189 Test Method for Conradson Carbon Residue of Petroleum Products
- D287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)
- D341 Practice for Viscosity-Temperature Charts for Liquid Petroleum Products
- D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
- D524 Test Method for Ramsbottom Carbon Residue of Petroleum Products
- D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration
- D974 Test Method for Acid and Base Number by Color-Indicator Titration
- D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
- D1401 Test Method for Water Separability of Petroleum Oils and Synthetic Fluids
- D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
- D2007 Test Method for Characteristic Groups in Rubber Extender and Processing Oils and Other Petroleum-Derived Oils by the Clay-Gel Absorption Chromatographic Method
- D2161 Practice for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

*A Summary of Changes section appears at the end of this standard

- D2270** Practice for Calculating Viscosity Index from Kinematic Viscosity at 40 and 100°C
- D2501** Test Method for Calculation of Viscosity-Gravity Constant (VGC) of Petroleum Oils
- D2622** Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry
- D2887** Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography
- D2896** Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration
- D3120** Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry
- D3339** Test Method for Acid Number of Petroleum Products by Semi-Micro Color Indicator Titration
- D3828** Test Methods for Flash Point by Small Scale Closed Cup Tester
- D4052** Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
- D4057** Practice for Manual Sampling of Petroleum and Petroleum Products
- D4059** Test Method for Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography
- D4175** Terminology Relating to Petroleum, Petroleum Products, and Lubricants
- D4177** Practice for Automatic Sampling of Petroleum and Petroleum Products
- D4291** Test Method for Trace Ethylene Glycol in Used Engine Oil
- D4294** Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry
- D4530** Test Method for Determination of Carbon Residue (Micro Method)
- D4628** Test Method for Analysis of Barium, Calcium, Magnesium, and Zinc in Unused Lubricating Oils by Atomic Absorption Spectrometry
- D4629** Test Method for Trace Nitrogen in Liquid Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection
- D4739** Test Method for Base Number Determination by Potentiometric Hydrochloric Acid Titration
- D4927** Test Methods for Elemental Analysis of Lubricant and Additive Components—Barium, Calcium, Phosphorus, Sulfur, and Zinc by Wavelength-Dispersive X-Ray Fluorescence Spectroscopy
- D4929** Test Methods for Determination of Organic Chloride Content in Crude Oil
- D4951** Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry
- D5185** Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
- D5293** Test Method for Apparent Viscosity of Engine Oils and Base Stocks Between -10°C and -35°C Using Cold-Cranking Simulator
- D5453** Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence
- D5762** Test Method for Nitrogen in Petroleum and Petroleum Products by Boat-Inlet Chemiluminescence
- D5800** Test Method for Evaporation Loss of Lubricating Oils by the Noack Method
- D5949** Test Method for Pour Point of Petroleum Products (Automatic Pressure Pulsing Method)
- D5950** Test Method for Pour Point of Petroleum Products (Automatic Tilt Method)
- D5984** Test Method for Semi-Quantitative Field Test Method for Base Number in New and Used Lubricants by Color-Indicator Titration
- D5985** Test Method for Pour Point of Petroleum Products (Rotational Method)
- D6160** Test Method for Determination of Polychlorinated Biphenyls (PCBs) in Waste Materials by Gas Chromatography
- D6304** Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration
- D6375** Test Method for Evaporation Loss of Lubricating Oils by Thermogravimetric Analyzer (TGA) Noack Method
- D6417** Test Method for Estimation of Engine Oil Volatility by Capillary Gas Chromatography
- D6443** Test Method for Determination of Calcium, Chlorine, Copper, Magnesium, Phosphorus, Sulfur, and Zinc in Unused Lubricating Oils and Additives by Wavelength Dispersive X-ray Fluorescence Spectrometry (Mathematical Correction Procedure)
- D6481** Test Method for Determination of Phosphorus, Sulfur, Calcium, and Zinc in Lubrication Oils by Energy Dispersive X-ray Fluorescence Spectroscopy
- D6749** Test Method for Pour Point of Petroleum Products (Automatic Air Pressure Method)
- D6822** Test Method for Density, Relative Density, and API Gravity of Crude Petroleum and Liquid Petroleum Products by Thermohydrometer Method
- D6892** Test Method for Pour Point of Petroleum Products (Robotic Tilt Method)
- D7042** Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity)
- D7094** Test Method for Flash Point by Modified Continuously Closed Cup (MCCCFP) Tester
- D7095** Test Method for Rapid Determination of Corrosiveness to Copper from Petroleum Products Using a Disposable Copper Foil Strip
- D7279** Test Method for Kinematic Viscosity of Transparent and Opaque Liquids by Automated Houillon Viscometer
- D7346** Test Method for No Flow Point and Pour Point of Petroleum Products
- D7419** Test Method for Determination of Total Aromatics and Total Saturates in Lube Basestocks by High Performance Liquid Chromatography (HPLC) with Refractive Index Detection

D7500 Test Method for Determination of Boiling Range Distribution of Distillates and Lubricating Base Oils—in Boiling Range from 100 °C to 735 °C by Gas Chromatography

D7751 Test Method for Determination of Additive Elements in Lubricating Oils by EDXRF Analysis

D7777 Test Method for Density, Relative Density, or API Gravity of Liquid Petroleum by Portable Digital Density Meter

E1687 Test Method for Determining Carcinogenic Potential of Virgin Base Oils in Metalworking Fluids

2.2 Government Standard:

EPA 8120 Chlorinated Hydrocarbons by GC/MS, EPA SW-846³

2.3 Other Standards:

IP 346 Determination of Polycyclic Aromatics in Unused Lubricating Base Oils and Asphaltene Free Petroleum Fractions—Dimethyl Sulphoxide Extraction Refractive Index Method⁴

IP 447 Petroleum Products—Determination of Sulfur Content—Wavelength-dispersive X-ray Fluorescence Spectrometry⁴

IP 510 Petroleum Products—Determination of Organic Halogen Content—Oxidative Microcoulometric Method⁴

3. Terminology

3.1 Definitions:

3.1.1 For definitions of standard terms used in this guide, see Terminology **D4175** or ASTM Dictionary of Engineering Science and Technology.

3.1.2 *base oil, n*—a base stock or a blend of two or more base stocks used to produce finished lubricants, usually in combination with additives.

3.1.3 *base stock, n*—a hydrocarbon lubricant component, other than an additive, that is produced by a single manufacturer to the same specifications (independent of feed source or manufacturer's location), and that is identified by a unique formula number or product identification number, or both.

3.1.4 *guide, n*—a series of options or instructions that do not recommend a specific course of action.

3.1.4.1 *Discussion*—Whereas a practice describes a general usage principle, a guide only suggests an approach. The purpose of a guide is to offer guidance, based on a consensus of viewpoints, but not to establish a fixed procedure. A guide is intended to increase the awareness of the user to available techniques in a given subject area and to provide information from which subsequent evaluation and standardization can be derived.

4. Significance and Use

4.1 Refinery and re-refinery feedstock and the processes to which the feed is subject determine the composition of the base stock produced. Once produced, other potential sources of variation include storage, transportation and blending. It follows that lubricating base oils will be of variable chemical

³ U.S. EPA, "Test Methods for Evaluating Solid Waste, Physical/Chemical," SW-846. Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

⁴ *Standard Methods for Analysis and Testing of Petroleum and Related Products*, Available from Energy Institute, London, 61 New Cavendish St., W1G 7AR, U.K., England. <http://www.energyinst.org>.

TABLE 1 Suggested Physical and Compositional Property Test Methods for Lubricant Base Oils^{A,B}

Property	Test Method
Physical properties	
Appearance	c
Color	D1500
Density at 15 °C, kg/m ³	D287, D1298, D4052, D6822, D7777
Flash point, °C	D92, D93, D3828, D7094
Kinematic viscosity at 40 °C and 100 °C, mm ² /s (cSt)	D445, D7042, D7279
Apparent viscosity between -5 °C and -35 °C	D5293
Viscosity-gravity constant	D2501
Pour point, °C	D97, D5949, D5950, D5985, D6749, D6892, D7346
Viscosity index	D2270
Volatility at 371 °C, percent off	D2887, D6417, D7500
Percent Evaporation loss	D5800, D6375
Water separability (demulsibility), 30 min, mL	D1401
Compositional properties	
Carbon residue, percent mass	D189, D524, D4530
Nitrogen, mg/kg	D4629, D5762
Precipitation number	D91
Saturates, mass percent	D2007, D7419
Sulfur, mass percent	D2622, D3120, D4294, D5453, IP 447

^A Specific application issues such as selection of tests, frequency of testing, and test levels are to be negotiated between the base oil buyer and the seller.

^B See **Appendix X2** for a discussion of each property.

^C Refer to **X2.1.1** for a discussion of this property.

composition. For this reason, characterization criteria for hydrocarbon lubricant base oils are frequently chosen from properties such as those listed in [Table 1](#) and/or [Table 2](#). If specification limits are established, they are usually related to the intended use of the base oil.

4.2 The consistent performance of hydrocarbon lubricant base oils is a critical factor in a wide variety of applications such as engine oils, industrial lubricants, and metalworking fluids. In addition, in many of these applications humans are exposed to the base oils as a component of a formulated product such that health or safety considerations may need to be addressed. This guide suggests a compilation of properties and potential contaminants that are understood by those knowledgeable in the manufacture and use of hydrocarbon lubricants to be significant in some or all applications. A discussion of each of the suggested properties and potential contaminants is provided in [Appendix X2](#), with each listed alphabetically within four categories.

4.3 The test methods, base oil properties, and potential contaminants suggested are those that would likely be useful in many common situations, although it is recognized that there are specific applications and situations that could have different requirements. Performance testing related to a specific application may serve as the basis for acceptability.

4.4 Issues such as frequency of testing and the specifics of how the test results are to be applied are not addressed in detail. It is the responsibility of the buyer and seller to determine and agree upon the implementation of this guide. This guide serves as a basis for that discussion.

5. Sampling

5.1 Sampling of lubricant base oils may be required as part of the buyer/seller arrangement. If a sampling program is required, sampling in accordance with Practice [D4057](#), [D4177](#), or a suitable alternative may be employed.

6. Procedure

6.1 Application of Guide:

6.1.1 This guide applies only to hydrocarbon lubricant base oils. Base oils containing detectable levels of esters, animal fats, vegetable oils, or other materials used as, or blended into, lubricants are not covered by this guide.

6.1.2 The frequency and extent of testing is to be determined based upon need. A property that can be shown to have minimal variation with time, a potential contaminant that can be shown to be consistently absent or at levels below concern, or a toxicological property that is shown to be satisfactory may justify infrequent testing or no additional testing. In such cases, reporting of typical expected values may be acceptable.

6.1.3 Some of the measurements could be performed on the individual base stocks, and then, knowing the test results and the proportions of the base stock components in the base oil, test values can be calculated. Similarly, laboratory blends of base stocks in appropriate ratios could substitute for actual stream samples when sampling is not practical. This procedure may be negotiated between the base oil buyer and the seller.

6.1.4 The test methods suggested are not an exhaustive list. Many nonstandardized methods are being used in the petroleum industry, such as supercritical fluid chromatography (SFC), and thin layer chromatography (TLC) methods for the

TABLE 2 Suggested Parameters for Contaminants and Toxicological Properties in Lubricant Base Oils^{A,B}

	Typical Levels	Test Method ^C
Chemical properties		
Acid number, mg KOH/g	≤0.10	D664 , D974 , D3339
Base number, mg KOH/g	≤0.30	D2896 , D4739 , D5984
Total chlorine, mg/kg	≤50	D4929 , D6443 , D7751 , IP 510
Copper corrosion, 3 h at 100 °C	1	D130 , D7095
Elemental analysis, mg/kg:		
Mg, Na, Ba, Cu, B, Pb, Mn, Ni, Si		D5185
Al, As, Cd, Ca, Fe, P, Zn, Cr, Sn,		(Also, D4628 , D4927 , D4951 , D6443 , D6481 , and D7751 have limited applicability)
Total of all above elements	≤25	
Glycol, mg/kg	≤5	D4291
PCB content, mg/kg	≤2	D4059 , D6160
Total volatile organic halogens, mg/kg	≤5	EPA 8120
Water, mg/kg	≤150	D6304
Toxicological properties		
Mutagenicity index	pass ^D	E1687
DMSO extractables, percent mass	pass ^D	IP 346
Long-term rodent carcinogenicity bioassay, number tumor-bearing animals/test group (percent)	pass ^D	^E

^A Specific application issues such as selection of tests, frequency of testing, and test levels are to be negotiated between the base oil buyer and the seller.

^B See [Appendix X2](#) for discussion of each property.

^C Local legislative and regulatory requirements may also apply when selecting the tests to be run.

^D For further information, see [Appendix X4](#).

^E Refer to [X4.1.1](#) for discussion on this test method.

determination of saturates content. Further, there are more complex tests available for some properties that might give equivalent or superior information. For example, estimates of dermal carcinogenic potential can be obtained from screener tests, such as Test Method E1687 or IP 346, but the Long-Term Rodent Carcinogenicity Bioassay (that is, mouse skin-painting assay) represents the definitive test for the determination of carcinogenicity hazard of base oils. It is up to the users of this guide to determine which test methods provide them with the information appropriate to their needs.

NOTE 1—Local legislative and regulatory requirements may also apply when selecting the tests to be run.

6.1.5 Some of the physical, compositional, and contaminant test methods cited in Table 1 and Table 2 are utilized outside of their published scopes. If this is the case, there typically is no other more appropriate method, and industry experience has shown the test method to give acceptable results.

6.1.6 If the test method is a modification to an accepted test method, it should be identified as such when providing information on a base oil (for example, DXXXX Mod.).

6.2 Properties and Potential Contaminants:

6.2.1 Table 1 and Table 2 contain suggested properties, potential contaminants, and commonly used test methods that one may want to include in a base oil evaluation.

6.2.2 Table 1 includes physical and compositional properties and test methods only.

6.2.3 Table 2 includes parameters that may relate to potential contaminants and to toxicological properties. Typical levels were compiled through a survey of base oil producers. For further details, see Research Report RR:D02-1416.⁵

6.2.4 A discussion of the significance of each property is provided in Appendix X2.

7. Keywords

7.1 base oil; base stock; hydrocarbon; lubricants; oil

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1416.

APPENDIXES

(Nonmandatory Information)

X1. SIGNIFICANCE OF ASTM GUIDE FOR CHARACTERIZING LUBRICANT BASE OILS

X1.1 *Rationale*—This guide was generated in response to a request from automobile manufacturers that ASTM Committee D02 develop a standard for re-refined base oils. As the document evolved through the consensus process, it was

agreed that it would be appropriate to present this information as an educational guide and to include base oils from various refining processes, including both re-refining of used oils and refining of crude oils.

X2. SIGNIFICANCE OF PROPERTIES OF LUBRICANT BASE OILS

X2.1 Physical Properties

X2.1.1 *Appearance*—Fully acceptable lubricant base oils are typically observed to be clear and bright. Simple visual inspection of lubricant base oils may indicate the absence or presence of undesirable contaminants (for example, water, haze wax, suspended materials, and so forth). If such contaminants are present, more definitive testing is recommended to assess their effect on other base oil or finished lubricant functional properties.

X2.1.2 *Color*—Determination of the color of lubricant base oils is used mainly for manufacturing control purposes and is an important 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 lubricant base oil. When the color range of a particular product is known, a variation outside the established range may indicate possible contamination with another product. It should be noted, however, that color is not always a reliable guide to product quality and should not be used indiscriminately in product specifications.

X2.1.3 *Density*—Accurate determination of density of petroleum products is necessary for the conversion of measured volumes to volumes at the standard temperature of 15 °C or 60 °F. These may then be converted to weight, mass, and/or bulk density using appropriate conversion tables. Accurate density determinations become critical with respect to transfer of custody.

X2.1.4 *Flash Point*—Safe operation of mechanical equipment requires an adequately high flash point. Flash point measures the tendency of the sample to form a flammable mixture with air under controlled laboratory conditions. Flash point can indicate the possible presence of highly volatile flammable materials in a relatively nonvolatile or nonflammable material, such as lubricant base oils.

X2.1.5 *Kinematic Viscosity*—Correct operation of equipment depends upon the appropriate viscosity of the liquid being used. Accurate measurement of the kinematic viscosity of lubricant base oils is essential and fundamental in the formulation of lubricants, ensuring that product specifications and performance capabilities can be met.

X2.1.5.1 Using appropriate viscosity conversion practices, for example Practice **D341** or Practice **D2161**, viscosities provided by different test methods or reported at different temperatures may be converted to kinematic viscosity at 40 °C and/or 100 °C if required.

X2.1.6 *Apparent Viscosity*—Using a direct-indicating, rotational viscometer, the cranking viscosity of base oil is typically measured to determine their suitability for use in engine oil formulations.

X2.1.7 *Viscosity-Gravity Constant (VGC)*—A useful function for the approximate characterization of hydrocarbon lubricating base oil. Values of VGC near 0.800 indicate samples of paraffinic character, while values close to 1.00 indicate a preponderance of aromatic structures.

X2.1.8 *Pour Point*—The pour point of a lubricant base oil is an indication of the lowest temperature of its utility for certain applications. The pour point is a function of the severity of the dewaxing operation employed in the refining process, and the chemical composition (normal paraffin content and carbon distribution) of the base oil.

X2.1.9 *Viscosity Index*—Viscosity index is an indicator of the variation in kinematic viscosity due to changes in temperature of a lubricant, indexed between 40 °C and 100 °C. A higher viscosity index indicates a smaller decrease in kinematic viscosity with increasing temperature of the lubricant.

X2.1.10 *Volatility*—Finished oil volatility is primarily a function of lubricant base oil volatility but can be influenced by the lubricant additives. Volatile organic compounds which evaporate from an engine crankcase may contribute to airborne hydrocarbon emissions as well as engine oil consumption.

X2.1.11 *Water Separability (Demulsibility)*—In service, lubricants are exposed to water contamination and turbulence, resulting in the formation of emulsions. The ability of the lubricant base oil to separate from water is critical to the successful formulation and performance of some lubricants. See **X2.3.9**, regarding water content, for related information.

X2.2 Compositional Properties

X2.2.1 *Carbon Residue*—Knowledge of the carbon residue, or coke- and ash-forming tendencies of lubricant base oils, provides an indication of suitability for high-temperature lubricant applications. In high-temperature applications, carbon residue can contribute to engine deposits and wear. High values of carbon residue indicate that a lubricant base oil may be unfit for this purpose.

X2.2.2 *Nitrogen Content*—Nitrogen is a naturally occurring element in crude petroleum and small quantities of nitrogen-containing compounds are frequently found in lubricant base oils. In addition, many lubricant additives contain nitrogen compounds. The concentration of nitrogen is often used as a measure of the presence of nitrogen-containing additives for quality purposes.

X2.2.3 *Precipitation Number*—The precipitation number is sometimes referred to in the industry as *asphaltenes*, since petroleum naphtha insolubles is the result reported. Low values of precipitation number are desirable because they provide an

indication that potentially reactive hydrocarbon insoluble materials are not present in the lubricant base oil. The presence of hydrocarbon insoluble material provides an indication of incomplete base oil processing.

X2.2.4 *Saturates Content*—Chemical composition can have an effect on the characteristics and performance capabilities of lubricant base oils in concert with formulation additives. The saturates level is a function of crude oil source, refining sequence, and refining process severity. Chromatographic test methods are used to measure saturates. Test Method **D2007** is known to require extended time to complete and may not provide reliable results for higher saturates materials.

X2.2.5 *Sulfur Content*—Sulfur is a naturally occurring element in crude petroleum and may act as an antioxidant in base oils. With respect to lubricant base oils, the sulfur content is a function of crude source, refining process, and processing severity. Knowledge of the presence of sulfur-containing compounds in lubricant base oils may also be important in predicting the potential for corrosion. See **X2.3.4**, regarding copper corrosion, for related information.

X2.3 Chemical Properties

X2.3.1 *Acid Number*—A low acid number for the lubricant base oil portion of formulated lubricants is necessary to minimize the potential for metal corrosion and to maximize the life of the system being lubricated. High values for acid number of lubricant base oils provide an indication that oxidation reaction by-products may be present which should have been neutralized or removed in the re-refining process.

X2.3.2 *Base Number*—A low base number for the lubricant base oil portion of formulated lubricants is necessary to ensure that oxidation reaction degradation products have been effectively neutralized or removed in the re-refining process.

X2.3.3 *Chlorine Content*—This is an indirect indication of contamination. See **X2.3.7** and **X2.3.8**, regarding PCB content and volatile organic halides, for related information.

X2.3.4 *Copper Corrosion*—Some sulfur compounds in lubricant base oils can have a corroding action on copper-containing metals and this corrosivity is not necessarily related directly to the total sulfur content. The effect can vary according to the chemical types of sulfur compounds present. The copper strip corrosion test is designed to assess the relative degree of corrosivity.

X2.3.5 *Elemental Analysis*—Lubricant base oils from a capable refining process are typically composed of carbon and hydrogen, with smaller concentrations of sulfur, nitrogen, and oxygen. Lubricant base oils should be essentially free of metallic elements. Sources of metallic elements potentially present in lubricant base oils include crude oil, refining or processing aids, residual lubricant additives, and residual corrosion or wear metals not removed in the re-refining process.

X2.3.6 *Glycol Content*—Engine oils can become contaminated with ethylene glycol from the engine coolant system during service. Used engine oils may be collected and re-refined into lubricant base oils. High glycol contents in

lubricant base oils indicate a deficiency in the re-refining process to adequately remove this contaminant.

X2.3.7 PCB Content—Lubricant base oils must be free of PCB (polychlorinated biphenyl) compounds to be suitable for use in commerce. Historically, it has been demonstrated that PCBs are not present in lubricant base oils manufactured from virgin crude petroleum sources. However, used PCB containing fluids may inadvertently be admixed with used lubricating fluids destined for re-refining into lubricant base oils.

X2.3.8 Volatile Organic Halides—Lubricant base oils should be essentially free of volatile organic halides because capable refining and re-refining processes would effectively remove all traces of these materials. The presence of volatile organic halides in lubricant base oils indicates contamination with chlorinated solvents normally used in metalworking fluids and automotive parts washing fluids, or improper transportation of base oils. See **X2.3.3**, regarding chlorine content, for related information.

X2.3.9 Water Content—Knowledge of the water content of lubricant base oils may be important to adequately determine emulsibility or demulsibility characteristics of formulated lubricants (see **X2.1.11** regarding water separability for related information) as well as concern for hydrolytic stability of additives and dielectric conductivity. Water is dissolved in all base oils at low concentrations when stored under atmospheric conditions in the presence of air; however, higher concentrations can occur when base oils are contaminated from external water sources.

X2.4 Toxicological Properties

X2.4.1 Mutagenicity Index (MI)—Test Method **E1687** is a microbiological screening technique based on a modification of the Ames (et al) Salmonella mutagenesis assay. It can be used as a screening technique to detect the dermal cancer hazard in lubricant base oils. The fundamental end point calculated from the data produced from this test method is the mutagenicity index (MI), which is used to predict the potential for base oils to elicit dermal carcinogenicity (**1,2**)⁶ Such a prediction should be evaluated on a case-by-case basis and may be more accurate when the MI and the results of an IP 346 test (see **X2.4.2**) are considered together. However, the standard mouse skin-painting bioassay (see **X2.4.3**) is the scientifically definitive test for such a prediction. See also **X4.1.3** for further information and discussion on test method limitations.

X2.4.2 Dimethyl Sulfoxide (DMSO) Extractables as defined by IP 346—This test concentrates and estimates polynuclear aromatic compounds (PAC), aromatic hydrocarbons, and related sulfur and nitrogen compounds, containing fused aromatic rings. These rings may have short alkyl or cyclo-alkyl

groups as substituents. Polynuclear aromatics are compounds that may be normally found in crude petroleum used oil and, to a lesser extent, in lubricant base oils; some of these materials have been shown to cause cancer. Minimum concentrations of these materials may be beneficial in formulated lubricants because they are believed to contribute natural oxidation stability, seal swell, and enhanced additive solubility characteristics.

X2.4.2.1 The IP 346 method is a gravimetric procedure in which a sample of oil is diluted with cyclohexane and extracted twice with DMSO. The sample is cut so as to exclude material boiling below 300 °C. The resulting extract includes such things as the 3-ring to 7-ring polycyclic aromatic hydrocarbons (some are known carcinogens) in the test sample but it is recognized that the method extracts other materials as well.

X2.4.2.2 The affinity of DMSO to specific hydrocarbon species increases as the number of aromatic rings increases, decreases as the length of the side chain increases, and is not selective between carcinogenic and noncarcinogenic PACs. This resulting DMSO extract is therefore larger, in terms of mass percent, than would be the case if the method extracted only carcinogens.

X2.4.2.3 Nevertheless, the percentage of DMSO extractables resulting from this test may be used to predict the potential for base oils to elicit dermal carcinogenicity and should be evaluated on a case-by-case basis (**3**). Such a prediction may be more accurate when the results are considered together with the results of Test Method **E1687** (see **X2.4.1**). However, the standard mouse skin-painting bioassay (see **X2.4.3**) is the scientifically definitive test for such a prediction. See **X4.1.2** for further information and discussion on test method limitations.

X2.4.3 Long-term Rodent Carcinogenicity Bioassay—This test, the standard mouse skin-painting bioassay, is the animal test model of choice to predict/assess the carcinogenicity hazard of petroleum products. Mice are dosed dermally with 25 µL to 100 µL of test material two to three times per week for a total duration ranging from 18 months to the lifetime of the animal. The development and characterization of tumors provides direct evidence of carcinogenicity hazard of a petroleum product. The time of tumor development provides an indication of the carcinogenic potency of the material. The mouse skin-painting bioassay is a sensitive indicator of polynuclear aromatic hydrocarbons, the carcinogenic moiety of lubricant base oils. Results in this test are determined based on the percentage of tumor-bearing animals in the treated groups compared with the percentage of tumor-bearing animals in the concurrent negative control groups as well as historical data on negative control animals. Analysis of the data should be performed on a case-by-case basis using sound scientific judgment and appropriate statistical analyses (**4,5**).

⁶ The boldface numbers refer to the list of references at the end of this standard.

X3. REGULATORY AND LEGISLATIVE CLASSIFICATION OF BASE STOCKS AND BASE OILS

X3.1 Carcinogenicity Classification of Base Stocks/Oils

X3.1.1 In various regions of the world there is existing or pending legislation covering the classification of hydrocarbon base stocks and base oils for carcinogenic hazard that may need to be considered in addition to, or in place of, some of the parameters provided in this guide.

X3.1.2 *United States (U.S.)*—In the U.S., the carcinogenicity classification of virgin base stocks is covered by 29 CFR Part 1910.1200 (6). For mineral oils, OSHA (Occupational Safety and Health Administration) base their classification on the results of the IARC (International Agency for Research on Cancer) review of the available carcinogenicity data on base stocks (7,8). Under the OSHA rules, base stocks are classified on the basis of their processing history. Thus, *mildly* hydrotreated or *mildly* solvent refined base stocks are considered to possess possible carcinogenic potential, whereas *severely* hydrotreated or *severely* solvent refined base stocks are not considered to present a carcinogenic hazard. OSHA has from time to time given interpretative guidance on the determination of processing severity (9). OSHA rule making clearly requires chemical manufacturers and importers to evaluate the hazards of the chemicals they produce or import.

X3.1.3 *European Union (EU)*—In EU countries, the carcinogenicity classification of base stocks is covered by the Regulation on the Classification, Labeling and Packaging of Substances and Mixtures (CLP) which entered into force in 2009 (10). The CLP Regulation replaces certain provisions of the directives related to the classification, packaging and labeling of dangerous substances. This classification system is based on the United Nations Globally Harmonized System of Classification and Labeling of Chemicals (GHS) also called the Purple Book (11, 12).

X3.1.3.1 For lubricating oils listed in CLP Annex VI, the CLP approach to distinguish between carcinogenic and non-carcinogenic mineral base oils is on the basis of oil refining process and chemical marker information (see X4.1.2.1). It divides base oils into three groups, based on the severity of the refining process: Unrefined or Mildly Refined Base Oils, Highly Refined Base Oils, and Other Lubricant Base Oils. (1) Unrefined or Mildly Refined Base Oils are classified in category 1A: substances known to have carcinogenic potential to humans. (2) Highly Refined Base Oils do not have a

classification in any of the carcinogenic categories. (3) Base oils that fall into the Other Lubricant Base Oils category contain a large number of individual CAS (Chemical Abstract Service) numbers with unspecified refining severity.

X3.1.3.2 It has been demonstrated (3) that it is possible to distinguish between carcinogenic and noncarcinogenic base oils on the basis of the level of DMSO extractables based on a correlation with the results from long-term rodent carcinogenicity bioassay studies using virgin base oils. As a result, the European Commission mandates that Method IP 346 be used as the basis for labeling certain petroleum refinery streams, including lubricating oil base stocks, for carcinogenicity. Oils with PAC content greater than 3 % (w/w) require labeling. Other data, such as results of long term skin painting tests or mutagenicity studies, cannot be used to override the classification (10).

X3.1.4 *Other*—The situation in the rest of the world is less clear as regulatory frameworks are still developing. There are, however, indications that regulatory authorities in other regions of the world are adopting, (with some variations), the GHS system of carcinogenicity classification. The UN Subcommittee of Experts on the Globally Harmonized System of Classification and Labeling of Chemicals (IPIECE) has provided guidance for classification of lubricating base oil that is similar to the EU approach of refining process and chemical marker (12).

X3.2 *Re-refined Oil*—Re-refined oil, as the term is used in commerce, is a generic term used to describe base oils derived from used oils subjected to various types and severities of treatment/processing and producing products of different composition. The potential human health effects of a specific distillate base stock derived from crude oil or used oil is inversely related to the severity and/or extent of processing the oil has undergone thus in most jurisdictions, base oil is classified by processing technique rather than feedstock (13). The issue of carcinogenic classification specific to re-refined base stocks has rarely been considered by regulators. There are some national technical specifications/guidelines for re-refined base stocks, but the parameters set out in these do not address health concerns or classification for possible carcinogenic potential (14, 15).

X4. ADDITIONAL INFORMATION ON TESTING OF BASE STOCKS AND BASE OILS FOR TOXICOLOGICAL PROPERTIES

X4.1 For those interested in a more detailed discussion of the current significance given to each of the toxicological tests identified in the guide, comments are provided on the use and scope each for determining the carcinogenic potential or classification of base stocks and base oils.

X4.1.1 *Long-term Rodent Carcinogenicity Bioassay*—This biological test is generally considered by regulatory authorities to be the definitive scientific test for determining the dermal carcinogenic potential of a material. Notwithstanding this, regulators recognize that the test is expensive to perform, requires a large number of animals, and takes a long time to obtain results. The oil industry has sought to develop alternative strategies for predicting the carcinogenicity of base stocks (16,17). While it is recognized that there are no formal regulatory guidelines on the conduct and interpretation of skin painting studies, the general principles recommended in these respects by OECD, US NTP and Horton, et. al. are usually followed (18, 19, 20).

X4.1.2 *Dimethyl Sulphoxide (DMSO) Extractables (IP 346)*—IP 346 is a standard analytical method developed by the Energy Institute (formerly Institute of Petroleum). Based on a correlation with long term skin painting data, the percentage mass of DMSO extractables can be used to predict the potential for virgin base stocks to elicit dermal carcinogenicity (21); there is no published correlation with re-refined base stocks/oils (18).

X4.1.2.1 The scope and limitations of the IP 346 method for carcinogenicity classification are defined by the IP methodology, the CONCAWE review, and the EU CLP(21,10). Briefly, the published scope and limitations are:

(1) The IP 346 method describes the determination of predominantly polycyclic aromatics (PAC) over the concentration range 1 % to 15 % by mass in unused, additive-free lubricating base oils (stocks), having an atmospheric boiling point of 300 °C min at 5 % recovered sample. This method may apply to PAC concentrations outside this range and to other asphaltene-free petroleum fractions, but the precision has not been determined.

(2) As the EU legislation is framed presently, the IP 346 marker only applies to individual lubricating base stocks and certain mixtures of base stocks, but not products containing additives (22).

(3) At present, there are insufficient data available to support the prediction of IP 346 content of base stocks/oils from other analytical parameters.

X4.1.3 *Mutagenicity Index (MI)—Test Method E1687 (Modified Ames)*—This is a short-term microbiological assay developed to detect the presence of mutagens in lubricant base stocks. It is based on the standard Salmonella mutagenesis assay but modified to enhance sensitivity to PAHs (polyaromatic hydrocarbons) in oils. Mutagenic potency also correlates with the content of 3-ring to 7-ring PACs, which include polycyclic aromatic hydrocarbons and their heterocyclic analogs. The strength of these correlations implies that PACs are the principal mutagenic and carcinogenic species in these oils. A correlation of mutagenicity with carcinogenicity was developed with results from this assay and those of long term skin painting data on virgin mineral oils; there is no published correlation with re-refined base stocks/oils (23, 24, 25). Guidance on protocols, interpretation and pass/fail criteria for modified Ames tests are included in the above referenced papers.

X4.1.3.1 The scope and limitations of the Modified Ames test are defined in Test Method E1687. Briefly the published scope and limitations are:

(1) The assay can be used as a method to detect the presence of potential dermal carcinogens in virgin base oils used in the formulation of metalworking fluid.

(2) The test method is designed to detect mutagenicity mediated predominantly by PNAs (polynuclear aromatics) derived from petroleum. It is recognized that the assay is disproportionately sensitive to nitroaromatic combustion products and as yet unidentified components of catalytically or thermally cracked stocks.

(3) The test method is not recommended as the sole testing procedure for oils which have viscosities less than 18 mm²/sec (90 SUS) at 40 °C, or for fully formulated metalworking fluids.

(4) Virgin oils with MI <1.0 have a high probability of being non-carcinogenic in a mouse skin-painting bioassay. Virgin oils with MI between 1.0 and 2.0 may or may not be carcinogenic in a mouse skin-painting bioassay. Whenever possible, corroborative data from PAC analyses or additional biological testing should be used in categorizing such oils for carcinogenic potential. It should also be understood that oils producing MIs close to the values separating categories may be indiscernibly different in a carcinogenicity assay from oils having MIs on the other side of that boundary.

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SUMMARY OF CHANGES

Subcommittee D02.P0 has identified the location of selected changes to this standard since the last issue (D6074 – 08) that may impact the use of this standard. (Approved April 1, 2015.)

- (1) *Introduction*—Moved first paragraph to **X1.1**; moved second and third paragraphs to Scope.
- (2) *Scope*—Added new **1.3** and the information from Introduction (above); renumbered the subsections.
- (3) *Referenced Documents*—Added 34 new references and removed 5 old references; footnotes adjusted accordingly; corrected title for IP 346 test method.
- (4) *Summary of Guide*—Removed and the next sections in the guide are renumbered.
- (5) *Significance and Use*—Added **4.1** and removed former subsection 5.2; renumbered all paragraphs.
- (6) *Sampling*—Added Practice **D4177**.
- (7) *Procedure*—Minor edits to **6.1.6** and **6.2.1**; added user caveat to **6.1.4**.

- (8) *Table 1*—Added Test Methods **D5293** and **D2501**; added 23 new test methods corresponding to properties already listed in the table; four test methods were removed. SI unit notations were corrected where required and footnotes were revised.
- (9) *Table 2*—Added 10 new test methods corresponding to properties already listed in the table; one test method was removed. SI unit notations were corrected where required and footnotes were revised.
- (10) *Appendixes*—Added new **Appendix X1**; renumbered and extensively updated **Appendix X2**, **Appendix X3**, and **Appendix X4**.
- (11) SI unit notations and usage was corrected.
- (12) All notes, footnotes and references are updated to match document revisions.

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