



# Standard Test Methods for Analysis of Lubricating Grease<sup>1</sup>

This standard is issued under the fixed designation D128; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the U.S. Department of Defense.*

<sup>ε1</sup> NOTE—A section reference in subsection 21.1 was corrected editorially in July 2015.

## 1. Scope

1.1 These test methods for analysis cover greases of the conventional type, which consist essentially of petroleum oil and soap. The constituents that can be determined are soap, unsaponifiable matter (petroleum oil, and so forth), water, free alkalinity, free fatty acid, fat, glycerin, and insolubles.

NOTE 1—Any of the test methods described herein are best used by an experienced grease analyst who may also be able to make appropriate adaptations of the techniques as occasion requires.

1.2 A supplementary test method is provided in **Appendix X1**. This test method is 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 nonpetroleum fluids or nonsoap-type thickeners, or both. In some cases, these constituents can react with strong acid or alkaline solutions.

1.3 These test methods appear in the following order:

	Sections
Ash Determination	7 – 11
Insolubles, Soap, Fat, Petroleum Oil, and Unsaponifiable Matter	12 – 20
Free Alkali and Free Acid	21 – 23
Water	24
Glycerin (Quantitative)	25 – 29

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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.*

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and are the direct responsibility of Subcommittee D02.G0.01 on Chemical and General Laboratory Tests.

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## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

- D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation
- D156 Test Method for Saybolt Color of Petroleum Products (Saybolt Chromometer Method)
- D217 Test Methods for Cone Penetration of Lubricating Grease
- D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
- D473 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method
- D804 Terminology Relating to Pine Chemicals, Including Tall Oil and Related Products
- D1078 Test Method for Distillation Range of Volatile Organic Liquids
- D1193 Specification for Reagent Water
- D1353 Test Method for Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products

## 3. Terminology

### 3.1 Definitions:

- 3.1.1 *asphalt, n*—a dark brown-to-black cementitious material in which the predominating constituents are bitumens.
  - 3.1.1.1 *Discussion*—Asphalt can be a natural product or a material obtained from petroleum processing.
- 3.1.2 *candle pitch, n*—a dark brown-to-black, tarry or solid, by-product residue from soap and candle stock manufacture, refining of vegetable oils, refining of wool grease, or refining of refuse animal fats.
- 3.1.3 *cup grease, n*—any lubricating grease having physical properties, such as consistency and texture, suitable for its use in spring-loaded or screw-type lubricating cups.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.1.3.1 *Discussion*—Cup greases are predominantly NLGI No. 3 or 4 calcium greases, but grease types other than calcium are also used.

3.1.4 *degras (wool fat, wool grease, wool wax)*, *n*—a fat-like material comprised primarily of sterols, other higher alcohols, and fatty acids, obtained from the solvent extraction of sheep's wool.

3.1.5 *free alkali*, *n*— in lubricating grease, unreacted basic (alkaline) material present in the product.

3.1.5.1 *Discussion*—Many greases are made with a slight excess of alkali to ensure complete saponification. Free alkali is determined by acidification of a solvent-thinned specimen and back titration with standardized, alcoholic potassium hydroxide. It is expressed in terms of the predominating alkali and a mass % of the total grease composition (for example, mass % lithium hydroxide).

3.1.6 *free fatty acid*, *n*—in lubricating grease, unreacted carboxylic acid(s) present in the product.

3.1.6.1 *Discussion*—Some greases are made with a slight excess of carboxylic acid to ensure a non-alkaline product. Free fatty acid is determined by neutralization of a solvent-thinned specimen with standardized, alcoholic potassium hydroxide. Regardless of the actual composition of the carboxylic acid(s), it is expressed as free oleic acid and as a mass % of the total grease composition.

3.1.7 *insolubles*, *n*—in lubricating greases analysis, the material remaining after the acid hydrolysis, water extraction, and solvent extraction of soap-thickened greases.

3.1.7.1 *Discussion*—Consisting of such products as graphite, molybdenum disulfide, insoluble polymers, and so forth.

3.1.8 *lubricating grease*, *n*—a semi-fluid to solid product of a dispersion of a thickener in a liquid lubricant.

3.1.8.1 *Discussion*—The qualifying term, *lubricating*, should always be used. The term, *grease*, used without the qualifier refers to a different product, namely certain natural or processed animal fats, such as tallow, lard, and so forth.

3.1.9 *mixed base*, *adj*—in lubricating grease, the description of a thickener system composed of soaps of two metals.

3.1.9.1 *Discussion*—Although mixed-base grease can be made with soaps of more than two metals, in practice, such is rarely, if ever, encountered. All of the soaps need not be thickeners, although the major soap constituent will be one capable of forming a lubricating grease structure. Because the mixed soaps are seldom present in equal amounts. The predominant soap is referred to first.

#### NLGI Lubricating Grease Guide<sup>3</sup>

3.1.10 *montan wax*, *n*—a wax-like material comprised primarily of montanic acid and its ester, higher aliphatic alcohols, and resins obtained from the solvent extraction of lignite.

3.1.11 *neutralization number*, *n*—of petroleum oil, the quantity of acid or base required to titrate to neutrality and

expressed as equivalent milligrams of potassium hydroxide per gram of sample. **D804**

3.1.12 *NLGI*, *n*—National Lubricating Grease Institute.

3.1.13 *NLGI number*, *n*—a numerical scale for classifying the consistency range of lubricating greases and based on the Test Methods **D217** worked penetration.

#### NLGI Lubricating Grease Guide

3.1.14 *non-soap thickener (synthetic thickener, inorganic thickener, organic thickener)*, *n*—in lubricating grease, any of several specially treated or synthetic materials, excepting metallic soaps, that can be thermally or mechanically dispersed in liquid lubricants to form the lubricating grease structure.

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3.1.15 *residuum*, *n*—a liquid or semi-liquid product obtained as residue from the distillation of petroleum and consisting primarily of asphaltic hydrocarbons.

3.1.15.1 *Discussion*—Also known as asphaltic oil, asphaltum oil, liquid asphalt, black oil, petroleum tailings, and residual oil.

3.1.16 *rosin oil*, *n*—a viscous, oily liquid obtained as a condensate when the residue (rosin) from turpentine production is subjected to dry, destructive distillation.

3.1.16.1 *Discussion*—Also used to describe specially compounded oils having a rosin base.

3.1.17 *saponification*, *n*—the interaction of fats, fatty acids, or esters generally with an alkali to form the metallic salt, which is commonly called soap.

3.1.17.1 *Discussion*—Soap thickeners are most often made by in situ saponification in the lubricating grease base oil. However, the use of pre-formed soaps is also common; dispersion is effected by mechanical means and usually with heat.

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3.1.18 *single base*, *adj*—in lubricating grease, relating to a thickener comprised of soaps of only one metal.

3.1.19 *soap*, *n*—in lubricating grease, a product formed in the saponification (neutralization) of fats, fatty acids, or esters by inorganic bases.

3.1.20 *Soxhlet apparatus*, *n*—a device, usually of glass, used to extract soluble material from a mixture of soluble and insoluble (generally solid) materials, by passing a volatile solvent through the sample and recirculating the solvent by refluxing.

3.1.21 *tar*, *n*—a brown or black, bituminous, liquid or semi-solid comprised primarily of bitumens condensed in the processing of coal, petroleum, oil-shale, wood, or other organic materials.

3.1.22 *thickener*, *n*—in lubricating grease, a substance composed of finely divided solid particles dispersed in a liquid lubricant to form the product's structure.

3.1.22.1 *Discussion*—The thickener can be fibers (such as various metallic soaps) or plates or spheres (such as certain non-soap thickeners), which are insoluble or, at most, only very slightly soluble in the liquid lubricant. The general requirements are that the solid particles be extremely small, uniformly

<sup>3</sup> Available from National Lubricating Grease Institute, 4635 Wyandotte St., Kansas City, MO 64112-1596.

dispersed, and capable of forming a relatively stable, gel-like structure with the liquid lubricant.

### D217

3.1.23 *thimble, n—in Soxhlet apparatus*, a closed-end porous cylinder used to hold the material to be extracted, usually made of thick matted filter paper but sometimes made of ceramic.

3.1.24 *total fluid constituent, n—in lubricating grease analysis*, the *n*-hexane-soluble material extracted from the lubricating grease sample.

3.1.24.1 *Discussion*—Typical materials include petroleum oil, non-petroleum fluid, soluble fats, and soluble additives.

3.1.25 *total n-hexane-insoluble material, n—in lubricating grease analysis*, that portion of grease (excluding free alkali) that is essentially insoluble in *n*-hexane.

3.1.25.1 *Discussion*—Typical materials include thickeners, fillers, inorganic salts, asphaltenes, or any combinations of these (also includes insoluble materials found in the analysis of contaminated grease). Free alkali content is generally insignificant.

3.1.26 *unsaponifiable matter, n—in lubricating grease*, organic materials, either added or found with fatty materials, which do not react during saponification.

## 4. Significance and Use

4.1 These test methods can be used to identify and estimate the amount of some of the constituents of lubricating greases. These test methods are applicable to many, but not all, greases.

4.2 Composition should not be considered as having any direct bearing on service performance unless such correlation is established.

NOTE 2—Details on other test methods for grease analysis can be found in other reference material.<sup>4,5,6</sup>

## 5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>7</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Type III.

<sup>4</sup> Stanton, G. M., "Examination of Grease by Infrared Spectroscopy," *NLGI Spokesman*, Vol 38, No. 5, August 1974, pp. 153–165.

<sup>5</sup> Stanton, G. M., "Grease Analysis, a Modern Multitechnique Approach," *Preprint NLGI Annual Meeting*, Oct 26–29, 1975.

<sup>6</sup> Bonomo, F. S., and Schmidt, J. J. E., "Development of Schematic Analytical Procedures for Synthetic Lubricants and Their Additives," *WADC Technical Report 54-464*, Part IV, July 1957 (U.S. Government No. AD-130922).

<sup>7</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

**TABLE 1 Physical Requirements of *n*-hexane**

Test	Requirement	ASTM Designation <sup>4</sup>
Initial boiling point, min, °C	66.1	D1078
Dry point, max, °C	68.9	D1078
Nonvolatile matter, max, g/100 mL	0.001	D1353
Color, Saybolt, min	+ 30	D156
Reaction with alkalis	A	...

<sup>4</sup> Boil 125 mL of *n*-hexane with 10 mL of 0.5 N alcoholic KOH solution and 50 mL of neutral 50 % alcohol for 1½ h on a hot plate. Use a glass tube about 7 mm in inside diameter and 750 mm in length as a reflux condenser. After cooling, titrate the solutions with 0.5 N HCl using phenolphthalein as the indicator. Not less than 9.8 mL of 0.5 N HCl shall be required for neutralization. The amount of alkali consumed in this test shall be deducted as a blank correction in the fat determination on Solution E.

5.3 *Acetone*—American Chemical Society Reagent Grade Acetone. (**Warning**—Extremely flammable. Vapors may cause flash fires.)

5.4 *Alcohol (50 %)*—The alcohol shall be prepared from commercial 95 % ethanol or denatured alcohol<sup>8,9</sup> (**Warning**—Flammable. Denatured. Cannot be made nontoxic) by distilling from NaOH and neutralizing exactly with NaOH or KOH using phenolphthalein as the indicator. (**Warning** —In addition to other precautions, avoid skin contact or ingestion.) Dilute with an equal volume of water.

5.5 *Ammonium Carbonate*—(NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>. (**Warning**—Harmful if swallowed.) (**Warning**—Harmful if swallowed.)

5.6 *Butter Yellow Indicator* (0.02 g/mL)—Toluene solution (**Warning**—Flammable. Vapor harmful) of *p*-dimethylaminoazobenzene. (**Warning**—Suspected carcinogen. In addition to other precautions, avoid inhalation or skin contact.)

5.7 *t-Butyl Alcohol*, melting point 24 °C to 25.5 °C (**Warning**—Flammable liquid; causes eye burns).

5.8 *Carbon Disulfide* (CS<sub>2</sub>). (**Warning**—Extremely flammable. Poison. Vapor may cause flash fire. Vapor harmful. Capable of self-ignition at 100 °C or above. Harmful or fatal if swallowed. May be absorbed through the skin.)

5.9 *Ethyl Ether*. (**Warning**—Extremely flammable. Harmful if inhaled. May cause eye injury. Effects may be delayed. May form explosive peroxides. Vapors may cause flash fire. Moderately toxic. Irritating to skin.)

5.10 *n-hexane*, high-purity grade,<sup>9,10</sup> conforming to the requirements of Table 1. (**Warning**—Extremely flammable. Harmful if inhaled. May produce nerve cell damage. Vapors may cause flash fire.)

<sup>8</sup> The sole source of supply of denatured grain alcohol known to the committee at this time is Formulas 1, 23-A, 30, and 35-A, as described in Publication No. 368, "Formulas for Denatured Alcohol," U.S. Treasury Dept., Internal Revenue Service.

<sup>9</sup> If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

<sup>10</sup> The sole source of supply of *n*-hexane, high-purity grade, known to the committee at this time is Phillips Petroleum Co., Special Products Div., Bartlesville, OK.

5.11 *Hydrochloric Acid (37 %)*—Concentrated acid (HCl). (**Warning**—Poison. Corrosive. May be fatal if swallowed. Liquid and vapor cause severe burns. Harmful if inhaled.)

5.12 *Hydrochloric Acid, Standard Solution (0.5 N)*—Prepare and standardize a 0.5 N solution of HCl.

5.13 *Hydrochloric Acid (10 %)*, a solution containing 10 % by weight of concentrated HCl in water, with a permissible variation of 0.5 %.

5.14 *Hydrochloric Acid (1 + 3)*—Mix one volume of concentrated HCl (37 %) with three volumes of water.

5.15 *Methyl Red Indicator Solution*. (**Warning**—Flammable. Avoid skin contact or ingestion.)

5.16 *Nitric Acid (1 + 4)*—Mix one volume of concentrated nitric acid (70 %) (**Warning**—Poison. Corrosive. Strong oxidizer. Contact with organic material may cause fire. May be harmful if swallowed. Liquid and vapor cause severe burns) with four volumes of water.

5.17 *Phenolphthalein Indicator Solution*—The phenolphthalein solution shall be prepared by dissolving 1.0 g phenolphthalein (**Warning**—In addition to other precautions, avoid skin contact or ingestion) in 50 mL of alcohol distilled as described in 5.4, adding 5 mL of water and neutralizing with NaOH or KOH.

5.18 *Potassium Hydrogen Sulfate (KHSO<sub>4</sub>)*. (**Warning**—Poison. Harmful or fatal if swallowed. Causes severe eye and skin irritation or injury. Dust or mist may be harmful.)

5.19 *Potassium Hydroxide (KOH), Alcoholic (0.5 N)*—Prepare and standardize a 0.5 N solution of alcoholic potassium hydroxide. (**Warning**—Poison. Causes eye and skin damage.)

5.20 *Potassium Periodate (KIO<sub>4</sub>)*. (**Warning**—May be irritating to skin and eyes. May react vigorously with reducing agents.)

5.21 *Sodium Carbonate*—(Na<sub>2</sub>CO<sub>3</sub>). (**Warning**—Harmful if swallowed. May cause skin irritation.)

5.22 *Sodium Hydroxide (240 g/L)*—Dissolve 240 g of sodium hydroxide (NaOH) (**Warning**—Poison. Causes eye and skin damage) in water and dilute to 1 L.

5.23 *Sodium Hydroxide Solution, Standard (0.05 N)*—Prepare and standardize a 0.05 N NaOH solution.

5.24 *Sulfuric Acid (1 + 4)*—Carefully mix one volume of concentrated sulfuric acid (H<sub>2</sub>SO<sub>4</sub> 95 %) with four volumes of water. (**Warning**—Poison. Corrosive. Strong oxidizer. Contact with organic material may cause fire. May be fatal if swallowed. Liquid and vapor cause severe burns. Harmful if inhaled. Contact with water liberates large amounts of heat.)

5.25 *Sulfuric Acid (3 + 20)*—Carefully mix 3 mL of concentrated sulfuric acid (95 %) with 20 mL of water.

5.26 *Toluene*. (**Warning**—Flammable. Vapor harmful.)

## 6. Sample

6.1 The sample size for the soap determination (Test Methods I and II for determination of insolubles, soap, fat, petroleum oil, and unsaponifiable matter) shall be from about 8 to 30

g, depending on the consistency of the grease, which is chiefly determined by the percentage of soap present. A 10 to 20-g sample is usually a convenient amount for No. 3 cup grease, while softer greases require a larger sample. The original sample shall be stirred or mixed until uniform.

6.2 Samples need not be weighed more closely than to 0.1 g.

## ASH DETERMINATION

### 7. General

7.1 The percentage of ash shall not be included in the total of the analysis.

NOTE 3—The determination of the total ash should not in general be regarded as of any great importance. It is, however, sometimes required. This determination is often unsatisfactory because of interaction between Na<sub>2</sub>CO<sub>3</sub> derived from the soap and inorganic insolubles. There is always likelihood of reaction with the porcelain crucible itself on account of the long continued heating necessary to burn off all carbon. Moreover, if much Na<sub>2</sub>CO<sub>3</sub> or K<sub>2</sub>CO<sub>3</sub> is present, the ash is fusible and often encloses carbon, making complete removal of the latter very difficult. Results will always be low in the presence of easily reducible oxides of volatile metals. There is also uncertainty as to when CaCO<sub>3</sub> has been completely ignited to CaO. Ash determinations made on the same sample in different laboratories are likely to vary widely.

### 8. Rapid Routine Test Method

8.1 Place a 2 g to 5 g sample of the grease in a weighed porcelain crucible and weigh the sample to the nearest 0.1 g. If lead or zinc soap is known to be absent, a platinum crucible is more convenient. Slowly burn off the combustible matter, and finally ignite the residue until the ash is free of carbonaceous matter. Cool the crucible and contents in a desiccator and weigh, reporting the result as percentage of ash.

### 9. Alternative Test Method

9.1 Burn the sample in accordance with 8.1 until the ash is nearly free from carbon. Cool the crucible and contents, dissolve the soluble portions in a little water, and add a slight excess of H<sub>2</sub>SO<sub>4</sub> (1 + 4), (**Warning**—Poison. Corrosive. Strong oxidizer. Contact with organic material may cause fire. May be fatal if swallowed. Liquid and vapor cause severe burns. Harmful if inhaled. Contact with water liberates large amounts of heat) running the acid in carefully from a pipet inserted under a small watch glass covering the crucible. Warm the crucible and contents on a steam bath until effervescence has ceased. Rinse the watch glass with water into the crucible. The solution, when tested with methyl red (**Warning**—Flammable. Avoid skin contact or ingestion) for free acidity, shall show free acid present. Evaporate the contents of the crucible to dryness and ignite the whole at a low red heat, adding a few small pieces of dry (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> (**Warning**—Harmful if swallowed) to drive off the excess SO<sub>3</sub>. After cooling and weighing, report the result as percentage of ash as sulfates.

NOTE 4—This test method gives more concordant results than the routine method (Section 8), but it requires more time and manipulation.

### 10. Qualitative Examination

10.1 An easily fusible ash, dissolving completely in water to give a strongly alkaline solution, indicates a grease containing

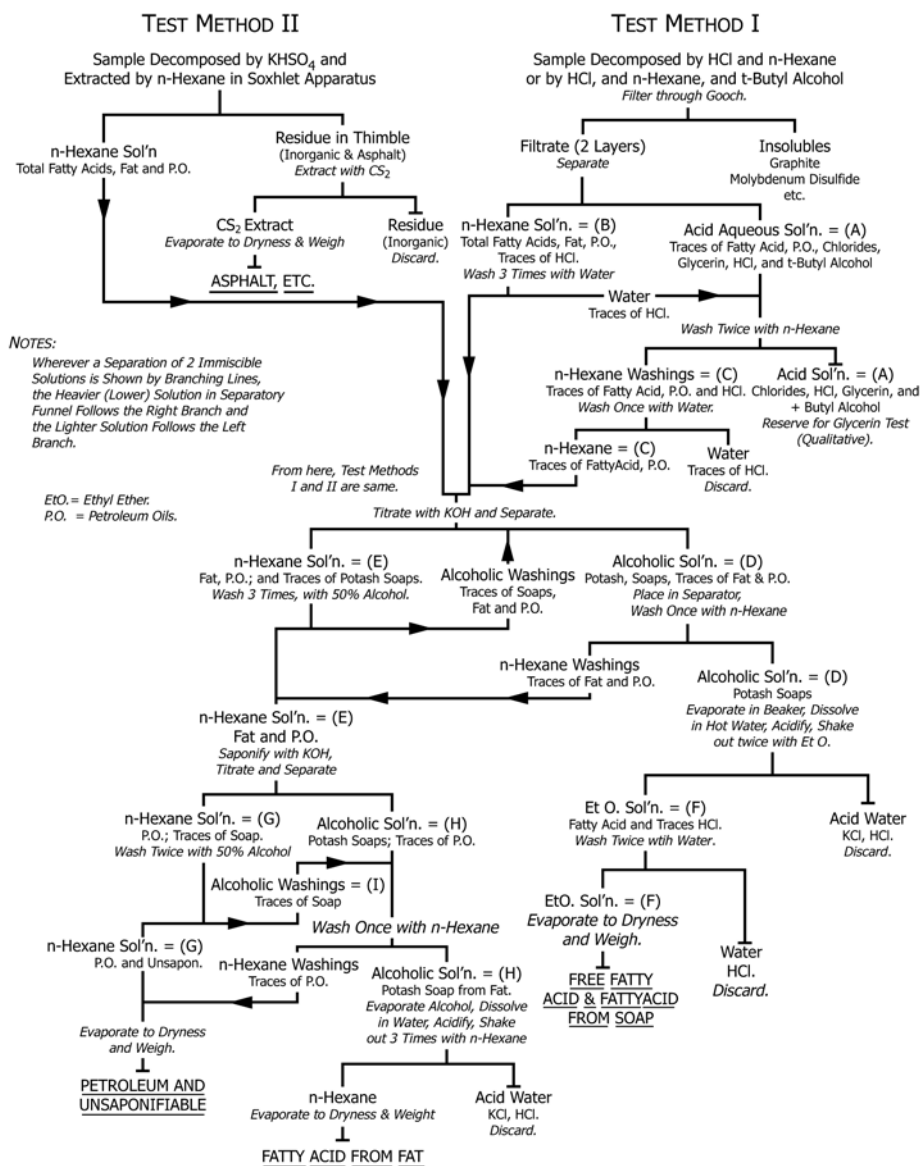


FIG. 1 Scheme of Grease Analysis

sodium, potassium, or both. Lithium is indicated by a white infusible ash, readily soluble in water, giving a strongly alkaline solution. A white infusible ash, practically insoluble in water but imparting to it an alkaline reaction, may indicate calcium, with or without magnesium or aluminum. Zinc is shown by the yellow color of the ash while hot, and lead may be indicated by the presence of metallic globules or by the yellow color of the ash when cold.

10.2 Dissolve the ash in  $\text{HNO}_3$  (1 + 4) (**Warning**—Poison. Corrosive. Strong oxidizer. Contact with organic material may cause fire. May be harmful if swallowed. Liquid and vapor cause severe burns) or HCl (1 + 3) (**Warning**—Poison. Corrosive. May be fatal if swallowed. Liquid and vapor cause severe burns. Harmful if inhaled.) Confirm the presence of the several bases by suitable chemical tests, following any standard scheme of qualitative analysis.

## 11. Quantitative Examination

11.1 For the quantitative examination of ash, use any standard analytical procedure, basing the choice of methods on the information gained from the qualitative tests. If only one base is present, a quantitative determination is, in general, unnecessary.

## INSOLUBLES, SOAP, FAT, PETROLEUM OIL, AND UNSAPONIFIABLE MATTER

### 12. Choice of Test Method

12.1 Examine greases containing residuum, asphaltic oils and asphalt, tars, and so forth, using Test Method II, as these ingredients usually cause stubborn emulsions if a vigorous agitation process is applied at the start. Analyze all other greases using Test Method I. (See Fig. 1.)

*Test Method I*
**13. Insolubles**

13.1 If insolubles are present (see **Note 5**), weigh the sample (see section **6**) in a small beaker, add 50 mL of HCl (10 %), and warm the beaker on a steam bath, stirring until all soap lumps have disappeared and the upper layer is clear. If undissolved mineral matter or other insoluble is present, filter both layers while warm through a Gooch crucible provided with a suitable mat, wash the beaker and crucible with warm (60 °C to 63 °C) water (see **Note 6**) and *n*-hexane, (**Warning**—Extremely flammable. Harmful if inhaled. May produce nerve cell damage. Vapors may cause flash fire.) Finally, wash the crucible with alcohol (**Warning**—Flammable. Denatured. Cannot be made nontoxic.) collecting the alcohol washings separately, and discarding them. Dry the crucible and contents at 120°C and weigh, reporting the result as percentage of insoluble matter (graphite, molybdenum disulfide, and so forth).

**NOTE 5**—If no insolubles are present, omit the procedure given in Section **13**.

**NOTE 6**—Throughout the test method where the word warm is used, it is to be understood that a temperature of 60 °C to 63°C is to be used.

**14. Soap**

14.1 If insolubles are absent, decompose a sample of the grease, either by the two-phase procedure as described in **14.2** or by the single-phase procedure as described in **14.3**. In either case, if the contents of the flask or beaker remain liquid, follow the procedure described in **14.4**. If contents congeal, or if, in the case of the two-phase decomposition, solid particles form, follow the procedure described in **14.5**.

14.2 Weigh the grease sample (see Section **6**) into either a 250 mL or a 500 mL Erlenmeyer flask, spreading the sample over the lower inside surfaces of the flask. Add 20 mL of *n*-hexane followed by 50 mL of HCl (10 %) and several boiling chips. Digest the sample by boiling under a reflux condenser until it is completely decomposed. Formation of a clear oil layer is evidence of complete decomposition. A digestion time of 3 h is generally sufficient; however, more vigorous boiling or use of a stronger HCl solution or additional *n*-hexane will be found necessary for greases difficult to decompose. Allow the contents of the flask to cool to room temperature.

14.3 Weigh the grease sample (see Section **6**) into a 250 mL beaker. Add a TFE-fluorocarbon-coated magnetic stirring bar, 50 mL of *t*-butyl alcohol (**Warning**—Flammable liquid; causes eye burns), 50 mL of *n*-hexane, 2 drops of butter yellow indicator (**Warning**—Flammable. Vapor harmful) and 2 mL of HCl (37 %). For heavy greases, 50 mL of toluene (**Warning**—Flammable. Vapor harmful) may be substituted for *n*-hexane. Note also that *t*-butyl alcohol will solidify if the temperature is approximately 24 °C to 27 °C or below. Heat the beaker and contents on a magnetic stirring hot plate. Stir magnetically and break up large lumps with a glass rod, but avoid boiling the solution. If the indicator becomes yellow, add concentrated HCl (37 %) in 1 mL increments until the color remains red. Continue the heating and stirring until all grease has dissolved, which indicates complete decomposition. For most grease,

10 min to 15 min are usually sufficient. Disregard any inorganic salts that may precipitate. Cool the contents of the beaker to room temperature.

14.4 Transfer the contents of the flask or beaker to a separatory funnel, using *n*-hexane and water as washing liquids. Allow this solution (or the combined filtrate and washings, except the alcohol washings, from the determination of insolubles, Section **13**, transferred to a separator), to clear and draw the aqueous layer, *A*, which contains all the bases as well as glycerin, into another separator. Wash the *n*-hexane layer, *B*, three times with 25 mL portions of water to remove HCl, adding the washings to *A*. Wash the aqueous solution, *A*, twice with 20 mL portions of *n*-hexane, *C*, and then set Solution *A* aside for examination for glycerin (Section **16**). Wash Solution *C* once with 15 mL of water, which may then be discarded, and add *C* to *B*. If Solutions *B* and *C* are comparatively light-colored, an approximate determination of free fatty acids and fatty acids from soap can now be made by titrating the solution in the separator with 0.5 *N* alcoholic KOH solution (**Warning**—Poison. Causes eye and skin damage) using phenolphthalein (**Warning**—In addition to other precautions, avoid skin contact or ingestion) as the indicator, and using 200 as the average neutralization value of the fatty acid (that is, 1.0 g of fatty acid requires 200 mg of absolute KOH for neutralization). If Solutions *B* and *C* are dark, add a few drops of phenolphthalein solution and sufficient 0.5 *N* alcoholic KOH solution to make the alcoholic layer distinctly alkaline after vigorous shaking.

**NOTE 7**—Capital letters reference solutions given in **Fig. 1**.

14.5 Heat the contents of the flask or beaker to approximately 63 °C and transfer to a separator that has been previously warmed by flushing with warm water, using warm *n*-hexane and warm water as washing liquids (**Warning**—see **14.5.1**). Allow this solution to clear and draw the aqueous layer, *A*, which contains all the bases as well as glycerin, into another separator. Wash the *n*-hexane layer, *B*, three times with 25 mL portions of warm water to remove HCl, adding the washings to *A*. Wash the aqueous solution, *A*, twice with 20 mL portions of warm *n*-hexane, *C*, and then set Solution *A* aside for examination for glycerin (see Section **16**). Wash Solution *C* once with 15 mL of warm water, which can then be discarded, and add *C* to *B*. If Solutions *B* and *C* are comparatively light-colored, an approximate determination of free fatty acids from soap can now be made by titrating the solution, which is at approximately 63 °C, in the separator with 0.5 *N* alcoholic KOH solution using phenolphthalein as the indicator, and using 200 as the average neutralization value of the fatty acid (that is, 1.0 g of fatty acid requires 200 mg of absolute KOH for neutralization). If Solutions *B* and *C* are dark, add a few drops of phenolphthalein solution and sufficient 0.5 *N* alcoholic KOH solution to make the alcoholic layer distinctly alkaline after vigorous shaking.

14.5.1 (**Warning**—Extreme care should be exercised in handling warm *n*-hexane because of pressure developing in the stoppered separatory funnel. Invert the funnel and release pressure through the stopcock occasionally.)

14.6 If Solutions *B* and *C* from 14.4 and 14.5 have been titrated, add 0.5 mL to 1.0 mL excess of alkali before separating. The conservative addition of alcohol at this point can aid in securing rapid and sharp separation. Allow the two solutions to separate sharply and draw off the lower alcoholic layer, *D*, into another separator. Wash the upper *n*-hexane layer, *E*, three times, with 30 mL, 25 mL, and 20 mL of neutral 50 % alcohol, respectively, adding these washes to *D*. Wash Solution *D* with 25 mL of *n*-hexane, after which draw off *D* into a beaker and add the *n*-hexane to *E*. Evaporate Solution *D* to a small volume to remove alcohol, wash the residue of potassium soap into a separator with hot water, acidify with HCl, and shake out twice with 50 mL and 25 mL of ethyl ether, *F*, respectively. Run Solution *F* into another separator and wash twice with 20 mL portions of water, which can then be discarded. Transfer Solution *F* to a weighed beaker and evaporate to dryness on a steam bath, blowing with air to remove all traces of ethyl ether. (**Warning**—Extremely flammable. Harmful if inhaled. May cause eye injury. Effects may be delayed. May form explosive peroxides. Vapors may cause flash fire. Moderately toxic. Irritating to skin.) Heat the residue, consisting of free fatty acids and fatty acids from soap, for a short time on a steam bath, adding and evaporating 5 mL portions of acetone (**Warning**—Extremely flammable. Vapors may cause flash fires) until a constant weight is obtained and the last traces of water are removed. Determine the exact neutralization value on as large a sample of these fatty acids as possible.

14.7 The fatty acids may be identified to some extent by special tests, such as odor, crystal form, melting point, iodine number, color reactions, and so forth.

14.8 If the grease is appreciably oxidized, the fatty acids obtained by the ethyl ether extraction are likely to be dark in color and hard to identify. For further study and identification, the neutralized acids may be extracted again (qualitatively) with *n*-hexane and HCl (1 + 3), thus eliminating the small amount of more darkly colored matter.

14.9 From the total quantity of fatty acid found, deduct the free acid, if any (see Section 23), and calculate the remainder as a percentage of soap as described in Section 15.

## 15. Calculation and Report

15.1 Calculate and report the neutralization number, molecular weight of fatty acids, and percentage of soap in accordance with 15.1.1 – 15.1.6:

15.1.1 *Neutralization Number of Fatty Acids*—Calculate the neutralization number of the fatty acids as follows:

$$A = 56.1BN/X \quad (1)$$

where:

*A* = neutralization number of fatty acids,  
*B* = millilitres of KOH solution required for titration of the solution,  
*N* = normality of KOH solution, and  
*X* = grams of fatty acid titrated.

15.1.2 *Molecular Weight of Fatty Acids*—Calculate the molecular weight of the fatty acids as follows:

$$M_f = 56,100/A \quad (2)$$

where:

*M<sub>f</sub>* = mean molecular weight of fatty acids, and  
*A* = neutralization number of fatty acids.

15.1.3 *Soap in Single-Base Greases*—Calculate the percentage of soap in single-base greases as follows:

$$\text{Soap, \%} = M_s C / M_f E \quad (3)$$

where:

*M<sub>s</sub>* = mean molecular weight of the soap,  
*C* = percentage of fatty acids from soap,  
*M<sub>f</sub>* = mean molecular weight of fatty acids from soap, and  
*E* = number of moles of fatty acid per mole of soap.

15.1.4 *Soap in Mixed-Base Greases Soluble in Organic Solvents*—If the grease is soluble in organic solvents, determine the more accurately determinable metal (of the two soaps present) in the solvent-soluble portion (see Note 8). Then calculate the percentage of the soap having this metal as the base, as follows:

$$\text{Soap, \%} = FM_s / M_m \quad (4)$$

where:

*F* = percentage of the metal determined,  
*M<sub>s</sub>* = mean molecular weight of the soap of the metal, and  
*M<sub>m</sub>* = molecular weight of the metal.

Calculate the fatty acids necessary to form this amount of normal soap. Subtract these acids from the total soap fatty acids and then calculate the remaining soap fatty acids to the soap of the second metal, as described in 15.1.3.

NOTE 8—The soluble portion may be separated from the insoluble matter by continuous extraction through an extraction thimble.

15.1.5 *Soap in Mixed-Base Greases Not Soluble in Organic Solvents*—If the grease is not soluble in organic solvents, determine the more accurately determinable metal and correct for free alkali if it is the predominant metal. Then proceed in accordance with 15.1.4. The presence of insolubles can introduce complications in the analysis of such a grease.

15.1.6 *Soap in Greases Containing Heavy Metal Soaps*—In greases containing soaps of lead, aluminum, and so forth, the soap content cannot be calculated accurately because free fatty acids, for which correction must be made, are not directly determinable in the presence of such soaps. A good estimate of the soap content may be obtained, however, by adding the value for organically combined metal to that for free fatty acids plus fatty acids (free and from soap) to soap. If the acids are greatly in excess of those necessary for the normal soap of the metal, it is advisable to calculate the metal to normal soap and report the remaining acids as excess acids.

## 16. Glycerin (Qualitative)

16.1 To determine whether a grease has been made from whole fats or from fatty acids, neutralize Solution A with dry Na<sub>2</sub>CO<sub>3</sub> (**Warning**—Harmful if swallowed. May cause skin irritation) and add sufficient excess to precipitate calcium or other metals. Then evaporate the whole mass to dryness, extract the residue several times with alcohol, filter the combined alcoholic extracts, and evaporate the alcohol. The residue will then contain most of the glycerin with a little

sodium chloride (NaCl). The presence or absence of glycerin in the residue shall be confirmed by suitable qualitative tests.

## 17. Fat

17.1 Concentrate the *n*-hexane solution, *E* (see Section 14), containing free fat, petroleum oils, and unsaponifiable matter to a volume of about 125 mL in a 300 mL Erlenmeyer flask, adding 10 mL of 0.5 *N* alcoholic KOH solution and 50 mL of neutral alcohol, and boil on a hot plate with an air condenser for 1½ h. Titrate the uncombined alkali with 0.5 *N* HCl, and from the alkali consumed, corrected for the blank determination as specified in 6.1, calculate the percentage of free fat, using 195 as the average saponification value (that is, 1.0 g of fat requires 195 mg of absolute KOH for specification).

17.2 Place the titrated solution, *G*, in a separator, draw off the alcoholic lower layer, *H*, into another separator, and remove the remaining traces of soap as described for Solutions *B* and *C* (see Section 14), making only two washes with 30 mL and 20 mL of 50 % alcohol, *I*. Combine Solutions *H* and *I*, wash once with a little *n*-hexane, which is added to Solution *G*, and then evaporate Solutions *H* and *I* to a small volume and isolate the fatty acid as described for *D* (see 14.6). The percentage of fat can be checked by weighing and titrating the free fatty acid.

NOTE 9—Multiplying the weight of fatty acid by 1.045 gives very close approximation of the weight of fat from which it was derived. This factor varies very little with the molecular weight of the fat.

## 18. Petroleum Oil

18.1 Evaporate the *n*-hexane solution, *G*, now containing all the hydrocarbon oils and unsaponifiable matter, to dryness in a weighed beaker as described for *F* (see 14.6), weigh the residue, and report the result as petroleum oils plus unsaponifiable matter.

18.2 The viscosity of the petroleum oil can be determined using Test Method D445. If a complete characterization of the petroleum products is required, decompose a new sample of from 150 g to 200 g of grease as described in Section 13, except that all quantitative operations shall be omitted, as well as the isolation of the free fatty acids, and the use of standard alkali and acid is not necessary.

18.3 If the grease contains rosin oil, beeswax, degreas, montan wax, or other materials containing a large amount of unsaponifiable substances, the petroleum oils isolated from Solution *G* will contain the unsaponifiable matter, and the physical properties will differ from those of the petroleum products used in making the grease. In most cases, no further separation is possible except in the hands of skillful and experienced operators, who can devise special methods to suit the individual conditions.

### Test Method II

## 19. Dark Greases

19.1 Weigh the sample in a 76 mm (3 in) porcelain dish, and add 10 g of granulated KHSO<sub>4</sub> (**Warning**—Poison. Harmful or fatal if swallowed. Causes severe eye and skin irritation or injury. Dust or mist may be harmful), 10 g of clean, dry ignited

sand, and 5 mL of water. Heat the dish and contents on a steam bath, while stirring frequently, until all water is driven off, 2 h usually being sufficient. After cooling and breaking up lumps with a small pestle, transfer the mixture quantitatively to an extraction thimble, which has been previously placed in a Soxhlet apparatus, using a little *n*-hexane to wash the last traces into the thimble. Extract the thimble thoroughly with *n*-hexane, concentrate the extract somewhat if necessary, and titrate the free fatty acid and fatty acid from soap with 0.5 *N* alcoholic KOH solution as in Solutions *B* and *C*, 14.4. Proceed in accordance with Sections 14, 17, and 18.

## 20. Asphalt and Tarry Matter

20.1 Extract the thimble a second time with CS<sub>2</sub> (**Warning**—Extremely flammable. Poison. Vapor may cause flash fire. Vapor harmful. Capable of self-ignition at 100 °C or above. Harmful or fatal if swallowed. May be absorbed through the skin.) Evaporate the extract to dryness, heat at 120 °C for 1 h, and then weigh, reporting the results as asphaltic and tarry matter. Discard the residue in the thimble.

### FREE ALKALI AND FREE ACID

## 21. Free Alkali

21.1 Weigh a 10 g to 30 g sample of the grease in a small beaker, dissolve as completely as possible in 75 mL of *n*-hexane by stirring with a spatula, wash the mixture into a 250 mL Erlenmeyer flask with a small amount of *n*-hexane, and rinse the beaker with 50 mL of 95 % alcohol (see 5.4), pouring the alcohol into the flask. Then add a few drops of phenolphthalein solution and shake the contents vigorously. If the alcoholic layer, after setting for a few seconds, is pink, add 10 mL of 0.5 *N* HCl, boil the solutions on a hot plate for 10 min to expel CO<sub>2</sub>, and titrate the excess acid back with 0.5 *N* alcoholic KOH solution. Calculate the free alkalinity in terms of hydroxide of the predominating base.

## 22. Insoluble Carbonates

22.1 If chalk or any other form of alkaline earth carbonate, or lead carbonate, is present as an insoluble, it will be detected by effervescence on adding the HCl. As the amount of such carbonates is likely to be considerable, increase the volume of 0.5 *N* HCl added sufficiently to dissolve all carbonate and leave a slight excess of acid. Boil the solution for 2 min, titrate the excess acid back with 0.5 *N* alcoholic KOH solution, and, from the acid consumed, calculate its equivalent in CaCO<sub>3</sub>, and so forth, disregarding any other forms of alkalinity that may have been present.

## 23. Free Acid

23.1 If the original alcoholic layer is not pink, titrate the unheated solution carefully with 0.5 *N* alcoholic KOH solution, shaking well after each addition. Calculate the acidity as oleic acid.

23.2 If soaps of iron, zinc, aluminum, or other weak bases are present, a determination of free acid is not possible since these metallic soaps react with KOH. Up to the present, no means has been devised whereby this determination can be made directly.



## WATER

### 24. Procedure

24.1 Determine water in accordance with Test Method **D95**. For solvent, use the petroleum distillate described in 5.1.2 of Test Method **D95**.

24.2 Calculate and report water as percent by weight in accordance with Test Method **D95**.

### GLYCERIN (QUANTITATIVE)

### 25. Scope

25.1 This test method is intended for the determination of free glycerin in grease and for glycerin present as fat in grease. The test method is applicable to greases containing 0.03 to 1.6 % of glycerin and is accurate even in the presence of ethylene and propylene glycols.

### 26. Summary of Test Method

26.1 To determine free glycerin in grease, the sample is refluxed with dilute sulfuric acid and *n*-hexane. The warm mixture is extracted with warm water, the washings neutralized and oxidized with potassium periodate,<sup>11</sup> (**Warning**—May be irritating to skin and eyes. May react vigorously with reducing agents) and the resulting solution titrated with standard sodium hydroxide solution (**Warning**—Poison. Causes eye and skin damage). Glycerin oxidizes to 2 mol of formaldehyde and 1 mol of formic acid, as follows:



26.2 To determine free glycerin and glycerin combined as fat, aqueous sodium hydroxide is added before refluxing, instead of sulfuric acid. After saponifying, the soaps are decomposed by refluxing with mineral acid, the *n*-hexane is evaporated, and the determination completed as for free glycerin.

#### *Determination of Free Glycerin*

### 27. Procedure

27.1 Weigh 10 g of the sample into a 250 mL Erlenmeyer flask and add dilute  $\text{H}_2\text{SO}_4$  (3 mL of concentrated  $\text{H}_2\text{SO}_4$  plus 20 mL of distilled water) and 20 mL of *n*-hexane.

27.2 Reflux the mixture on a steam bath until the grease is disintegrated. Allow the contents of the flask to cool to room temperature. If contents remain liquid, follow the procedure prescribed in **27.3**. If contents congeal or if solid particles form, follow the procedure prescribed in **27.4**.

27.3 Add 50 mL of *n*-hexane and transfer the contents of the flask quantitatively to a 500 mL separator, washing the flask with 50 mL of *n*-hexane and 25 mL of distilled water, and introducing these washings into the separator. Shake the contents of the separator and allow the two layers to separate. Draw off the water layer into a second separator containing

100 mL of *n*-hexane. Wash the first separator with two 30 mL portions of distilled water and add these washings to the second separator.

27.4 Heat contents of the flask to approximately 63 °C and transfer quantitatively to a 500 mL separator, washing the flask with 50 mL of warm *n*-hexane and 25 mL of warm distilled water, and introducing these washings into the separator. Keep the contents of the separator warm, shake, and allow the two layers to separate. Draw off the water layer into a second separator containing 100 mL of *n*-hexane. Wash the first separator with two 30 mL portions of warm distilled water and add these washings to the second separator.

27.5 Shake the second separator, allow the contents to separate, and filter the water layer into a 300 mL ground-glass stoppered Erlenmeyer flask. Wash the second separator once with 20 mL of distilled water and filter the washings into the same Erlenmeyer flask. The *n*-hexane solutions may then be discarded.

27.6 Add three drops of methyl red indicator solution (see **Note 10**) to the aqueous solution and make almost neutral with NaOH solution (240 g/L). Add two more drops of the indicator solution and carefully titrate the solution to a copper-colored end point with 0.05 *N* NaOH solution.

**NOTE 10**—The indicator is red in a neutral or acid solution. An alkaline (aqueous unadulterated) solution is yellow. In the glycerin determination the solution is copper-colored, corresponding to faint alkalinity.

27.7 *Blank*—Prepare a blank by adding 23 mL of  $\text{H}_2\text{SO}_4$  (3 + 20) to 80 mL of distilled water in a 300 mL glass-stoppered Erlenmeyer flask, and neutralize the solution to the methyl red end point as described in **27.6**.

27.8 Add 3 g of cp  $\text{KIO}_4$  to each flask. Tie the stoppers carefully in place and shake the flasks for 2 h. (A mechanical shaker is recommended for this purpose.) Remove excess  $\text{KIO}_4$  by filtering the solutions through close-textured paper and collect the filtrates in clean, 300 mL, glass-stoppered Erlenmeyer flasks. Add six drops of methyl red indicator solution to each flask and titrate the solutions to a copper-colored end point with 0.05 *N* NaOH solution. Record the amount of NaOH solution used.

#### *Determination of Total Glycerin (Free and Combined)*

### 28. Procedure

28.1 Weigh 10 g of the sample into a 250 mL Erlenmeyer flask. Add 10 mL of NaOH (100 g/L) and heat the mixture under a reflux condenser for 2 h.

28.2 Cool the flask and substantially neutralize the NaOH with  $\text{H}_2\text{SO}_4$  (1 + 4); then add an excess of approximately 25 mL of  $\text{H}_2\text{SO}_4$  (1 + 4) followed by 20 mL of *n*-hexane.

28.3 Complete the determination in accordance with **27.2 – 27.8**.

### 29. Calculation

29.1 Calculate the percentage of glycerin as follows:

$$G = \frac{(M - M_1) \times N \times 0.0921}{S \times 0.92} \times 100 \quad (6)$$

<sup>11</sup> Hoepe, G., and Treadwell, W. D., *Chemical Abstracts*, Vol 36, p. 4058.

where:

- G* = percentage of glycerin.  
*M* = millilitres of 0.05 N NaOH solution used to titrate the sample,  
*M<sub>l</sub>* = millilitres of 0.05 N NaOH solution used to titrate the blank,  
*N* = normality of the NaOH solution,  
*S* = grams of sample used, and  
0.92 = an empirical factor derived from experimental data (not to be confused with 0.0921, which is the stoichiometric glycerin value).

### 30. Precision and Bias

30.1 *Precision*—There are no interlaboratory test data available to establish a statistical statement of precision.

30.2 *Bias*—There are no interlaboratory test data available to establish a statistical statement on bias.

### 31. Keywords

31.1 analysis; extraction; fat content; fluid content; free alkali content; free fatty acid content; glycerin content; insolubles content; lubricating grease; petroleum oil content; soap content; Soxhlet apparatus; thickener content; water content

## APPENDIX

### (Nonmandatory Information)

#### X1. SUPPLEMENTARY TEST METHOD FOR THE ANALYSIS OF LUBRICATING GREASES

##### X1.1 Scope

X1.1.1 This test method includes procedures for the determination of the total fluid constituent and the total *n*-hexane-insoluble material. It is primarily intended for application to greases of the types discussed in 1.2.

NOTE X1.1—The determination of free fatty acid (see Section 23), and free alkali (see Section 21) are also involved in these procedures, since their respective test results are involved as correction terms in the calculations.

##### X1.2 Summary of Test Method

X1.2.1 *Total Fluid Constituent* (Note X1.2)—The grease is extracted with *n*-hexane. The resultant *n*-hexane-soluble fraction, when corrected for free fatty acids, is designated as total fluid constituent.

NOTE X1.2—Fats that are *n*-hexane-soluble will be included in the total fluid constituent but usually are not present in sufficient quantity to seriously affect the total fluid constituent properties.

X1.2.2 *Total n-hexane-Insoluble Material*—The *n*-hexane insoluble portion of the grease minus any free alkali is reported.

##### X1.3 Terminology

X1.3.1 *total fluid constituent*—the mineral oil, or nonpetroleum fluid and *n*-hexane-soluble fats, or all three.

X1.3.2 *total n-hexane-insoluble material*—soap, nonsoap thickeners, fillers, inorganic salts from free alkali, asphaltenes, or any combination of these, essentially insoluble in *n*-hexane.

##### X1.4 Apparatus

X1.4.1 *Extraction Apparatus*, conforming to requirements given in Test Method D473. A 1000 mL flask shall be used. A double-thickness filter paper extraction thimble, 33 mm by 94 mm, shall be suspended by corrosion-resistant wire or by a

corrosion-resistant wire basket below the condenser in such a position that the condensed solvent will enter the thimble.

##### X1.5 Procedure

X1.5.1 Weigh to the nearest 1 mg 10 g  $\pm$  0.2 g (see Note X1.3) of a representative sample into the *n*-hexane-washed, dried, tared extraction thimble, and place the thimble in the extraction apparatus to which 125 mL of *n*-hexane (see 5.10) has previously been added. Place the assembly on a steam plate (see Note X1.4) and extract for 12 h (see Note X1.5). Remove the thimble from the apparatus, dry the insoluble material for 30 min in a convection oven at 100 °C, and record the weight (see Note X1.3). To ensure complete removal of fluid constituent, re-extract the insoluble material for 1 h. Dry and reweigh (see Note X1.3). If the weight loss is greater than 20 mg, re-extract for 4 h more. Repeat the drying operation.

NOTE X1.3—A weighing bottle<sup>9,12</sup> should be used in this operation to minimize the absorption of moisture by the extraction thimble. The extraction is facilitated by spreading the grease sample uniformly to within 10 mm from the top of the thimble.

NOTE X1.4—As a safety precaution, a steam plate or other suitable safe heating device should be used for heating the solvent during the extraction step and for evaporating the solvent from the extracted fluid constituent.

NOTE X1.5—The reflux rate should be such that the solvent level in the extraction thimble is maintained between the top surface of the grease and the top of the thimble. It may be necessary to insulate the extraction flask from the steam plate to obtain a satisfactory reflux rate. As the extraction progresses, it may then be necessary to remove at least part of the insulation to increase the reflux rate in order to keep the extraction thimble full of solvent.

X1.5.2 If the *n*-hexane-soluble portion is not clear, indicating the presence of *n*-hexane-insoluble material or free acid, or both, filter while warm through a weighed, chemically

<sup>12</sup> The sole source of supply of the weighing bottle of suitable size known to the committee at this time is Catalog No. 15840, New York Laboratory Supply Co., 510 Hempstead Turnpike, West Hempstead, NY 11552.

resistant, medium-porosity fritted-glass crucible (for example, borosilicate glass). With hot *n*-hexane, transfer the remaining residue from the extraction flask into the glass crucible and then wash the crucible and contents with hot *n*-hexane to remove all traces of fluid constituent. This will remove the insoluble material while the free acid will remain in the *n*-hexane solution. The free acids may recrystallize on cooling. Dry the crucible and contents for 30 min at 100 °C in a convection oven. Cool and reweigh. This amount of material should be added to the weight of the total *n*-hexane-insoluble material.

X1.5.3 Transfer the *n*-hexane filtrate to a tared beaker. Evaporate the solvent on the steam plate with a jet of air or inert gas impinging on the surface for 3 h, and place in a convection oven for 1 h at 100 °C. Heat to constant weight.

NOTE X1.6—A 5 mg weight loss after a 15 min heating period in the oven can be considered to be constant weight.

## X1.6 Calculations

X1.6.1 Calculate the percentage of the total fluid constituent as follows:

$$\text{Total fluid constituent, \%} = (100 A/W) - B \quad (\text{X1.1})$$

where:

- A = *n*-hexane-soluble residue, g, (X1.5.3),
- W = sample used, g, and
- B = percentage of free fatty acid as oleic (Section 23).

X1.6.2 Calculate the percentage of total *n*-hexane-insoluble material as follows:

$$\text{Total } n\text{-hexane-insoluble material, \%} = (100C/W) - D \quad (\text{X1.2})$$

where:

- C = residue from *n*-hexane extraction, g, (X1.5.1),
- W = sample used, g, and
- D = percentage of free alkali as the hydroxide of the predominating base (Section 21).

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