Designation: D128 – 98 (Reapproved 2019)

Standard Test Methods for Analysis of Lubricating Grease[1]

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.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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.

[1] 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.

Current edition approved July 1, 2019. Published August 2019. Originally approved in 1922. Last previous edition approved in 2014 as D128 – 98 (2014)ε1. DOI: 10.1520/D0128-98R19.

[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.

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.

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[3]

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.

NLGI Lubricating Grease Guide

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. **NLGI Lubricating Grease Guide**

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

[3] Available from National Lubricating Grease Institute, 4635 Wyandotte St., Kansas City, MO 64112-1596; http://www.nlgi.org.

slightly soluble in the liquid lubricant. The general requirements are that the solid particles be extremely small, uniformly 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.[4,5,6]

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.[7] 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.

TABLE 1 Physical Requirements of *n*-hexane

Test	Requirement	ASTM Designation[A]
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 alkalies	[A]	...

[A] 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*.

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

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[8,9] (**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*—$(NH_4)_2CO_3$. (**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_2). (**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,[9,10] conforming to the requirements of Table 1. (**Warning—**Extremely flammable.

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

[5] Stanton, G. M., "Grease Analysis, a Modern Multitechnique Approach," *Preprint NLGI Annual Meeting*, Oct 26–29, 1975.

[6] 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).

[7] *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.

[8] 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.

[9] 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,[1] which you may attend.

[10] 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.