Designation: ~~D6074 – 08~~ D6074 – 15

Standard Guide for Characterizing Hydrocarbon Lubricant Base Oils[1]

This standard is issued under the fixed designation D6074; 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.

~~**INTRODUCTION**~~

~~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.~~

~~This guide 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.~~

~~This guide does not intend to cover all base oil viscosity grades. However, it does cover the majority of viscosities that would be used in both automotive and industrial oil formulations.~~

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.~~ 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~~2 mm^2/s to 40 mm^2/s (cSt) at ~~100°C (50 to 3740 SUS at 100°F).~~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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D91 Test Method for Precipitation Number of Lubricating Oils

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

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[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**

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)
~~D1744 Test Method for Determination of Water in Liquid Petroleum Products by Karl Fischer Reagent~~
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
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)
~~D5480~~D5293 Test Method for ~~Engine Oil Volatility by Gas Chromatography~~Apparent Viscosity of Engine Oils and Base Stocks Between –10 °C and –35 °C Using Cold-Cranking Simulator ~~(Withdrawn 2003)~~
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-846[3]

2.3 *Other Standards:*

IP 346 Determination of Polycyclic Aromatics ~~and Other Species in Petroleum Fractions by Dimethyl Sulfoxide—Refractive~~in Unused Lubricating Base Oils and Asphaltene Free Petroleum Fractions—Dimethyl Sulphoxide Extraction Refractive Index Method[4]

~~CEC L-40-A-93~~IP 447 ~~Evaporation Loss of Lubricating Oils (NOACK)~~Petroleum Products—Determination of Sulfur Content—Wavelength-dispersive X-ray Fluorescence Spectrometry[4]

~~JPI-5S-41-93~~IP 510 ~~Method B, Determination of Evaporation Loss of Engine Oils (Unified NOACK)~~Petroleum Products—Determination of Organic Halogen Content—Oxidative Microcoulometric Method[4]

~~29 CFR Part 1910 Hazard Communication; Interpretation Regarding Lubricity Oils, Federal Register, Part 50 (245), pp. 5182–5185~~[8]

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] 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*, ~~Vol 2.~~ Available from Energy Institute, London, 61 New Cavendish St., ~~W.I., England.~~W1G 7AR, U.K., England.http://www.energyinst.org.

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. Summary of Guide

4.1 This guide suggests a listing of properties and potential contaminants whose determination may be important for a hydrocarbon base oil due to performance, regulatory, or other considerations. Specific application issues such as frequency of testing and the use of other test methods are addressed only in a qualitative manner.

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 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 ~~of significance~~ significant in some or all applications. A discussion of each of the suggested properties and potential contaminants is provided in Appendix ~~X1~~X2, with each listed alphabetically within four categories.

5.2 Potential sources of base oil variation include the raw material, manufacturing process, operating conditions, storage, transportation, and blending.

TABLE 1 Suggested Physical and Compositional Property Test Methods for Lubricant Base Oils[A,B]

colwidth="1.47in"/COLSPEC

Property	Test Method
Physical properties	
Appearance	[C]
Color	D1500
~~Density at 15°C, kg/m^3~~	~~D1298, D4052~~
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~~
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, % off~~	~~D2887, D5480~~
Volatility at 371 °C, percent off	D2887, D6417, D7500
~~% Evaporation loss~~	~~NOACK (CEC L-40-A-93 or JPI-5S-41-93)~~
Percent Evaporation loss	D5800, D6375
Water separability (demulsibility), 30 min, mL	D1401
Compositional properties	
~~Carbon residue, % mass~~	~~D524, D189, D4530~~
Carbon residue, percent mass	D189, D524, D4530
Nitrogen, mg/kg	D4629, D5762
Precipitation number	D91
~~Saturates, wt %~~	~~D2007~~
Saturates, mass percent	D2007, D7419
~~Sulfur, wt %~~	~~D2622, D4294, D3120~~
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 ~~X1~~X2 for a discussion of each property.
[C] Refer to ~~X1.1.1~~X2.1.1 for a discussion of this property.

TABLE 2 Suggested Parameters for Contaminants and Toxicological Properties in Lubricant Base Oils[A,B]

colwidth="1.52in"/COLSPEC	Typical Levels	Test Method[C]
Chemical properties		
Acid number, mg KOH/g	≤0.10	D974, D664
Acid number, mg KOH/g	≤0.10	D664, D974, D3339
Base number, mg KOH/g	≤0.30	D4739, D2896
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
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,
Total of all above elements	≤25	havelimited applicability)
Total of all above elements	≤25	D6481, and D7751 have limited applicability)
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	D1744
Water, mg/kg	≤150	D6304
Toxicological properties[C ,D,E]		
Toxicological properties		
Mutagenicity index	pass[D]	E1687
DMSO extractables, wt %	pass	IP 346
DMSO extractables, percent mass	pass[D]	IP 346
Chronic animal bioassay analysis, number tumor-bearing animals/test group (%)	pass[F]	[G]
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 X1X2 for discussion of each property.

[C] Chronic animal bioassay analysis (that is, mouse skin-painting assay) represents the definitive test for the determination of potential carcinogenicity of base oils. Estimates of dermal carcinogenic potential can be obtained for virgin base oils from *screener* tests, such as Test Method E1687 or IP 346. There presently are no published chronic skin-painting studies with re-refined base oils.

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

[D] For further information, see Appendix X2X4.

[F] Passing results are 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 groups. Analysis of the data should be performed on a case-by-case basis using sound scientific judgment and appropriate statistical analyses.

[E] Refer to X1.4.3X4.1.1 for discussion on this test method.

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 thea specific application shouldmay 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 ~~should~~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 ~~high-performance liquid chromatography (HPLC),~~ supercritical fluid chromatography (SFC), and thin layer chromatography (TLC) methods for the 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 ~~Chronic Animal Bioassay Analysis~~ Long-Term Rodent Carcinogenicity Bioassay (that is, mouse ~~skin painting~~ 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~~ : Contaminants:*

6.2.1 ~~The~~Table 1 ~~following tables~~and Table 2 contain suggested properties, potential contaminants, and commonly used test methods that one ~~might~~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 ~~X1~~X2.

7. Keywords

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

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.

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