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An American National Standard

Standard Guide for Characterizing Hydrocarbon Lubricant Base Oils[1]

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INTRODUCTION

This guide is 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 purport to cover all tests which could be employed. 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 This guide is relevant to base oils composed of hydrocarbons and intended for use in formulating products including automotive and industrial lubricants. These base oils would typically have a viscosity of approximately 2 to 40 mm^2/s (cSt) at 100°C (50 to 3740 SUS at 100°F).

1.4 *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]

D 91 Test Method for Precipitation Number of Lubricating Oils

D 92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester

D 97 Test Method for Pour Point of Petroleum Products

D 130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test

D 189 Test Method for Conradson Carbon Residue of Petroleum Products

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)

D 524 Test Method for Ramsbottom Carbon Residue of Petroleum Products

D 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration

D 974 Test Method for Acid and Base Number by Color-Indicator Titration

D 1298 Practice for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

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

D 1401 Test Method for Water Separability of Petroleum Oils and Synthetic Fluids
D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
D 1744 Test Method for Water in Liquid Petroleum Products by Karl Fischer Reagent[3]
D 2007 Test Method for Characteristic Groups in Rubber Extender and Processing Oils and Other Petroleum-Derived Oils by the Clay-Gel Absorption Chromatographic Method
D 2270 Practice for Calculating Viscosity Index from Kinematic Viscosity at 40 and 100°C
D 2622 Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry
D 2887 Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography
D 2896 Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration
D 3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry
D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D 4059 Test Method for Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography
D 4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants
D 4291 Test Method for Trace Ethylene Glycol in Used Engine Oil
D 4294 Test Method for Sulfur in Petroleum and Petroleum Products by Energy-Dispersive X-Ray Fluorescence Spectrometry
D 4530 Test Method for Determination of Carbon Residue (Micro Method)
D 4628 Test Method for Analysis of Barium, Calcium, Magnesium, and Zinc in Unused Lubricating Oils by Atomic Absorption Spectrometry
D 4629 Test Method for Trace Nitrogen in Liquid Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection
D 4739 Test Method for Base Number Determination by Potentiometric Titration
D 4927 Test Methods for Elemental Analysis of Lubricant and Additive Components—Barium, Calcium, Phosphorus, Sulfur, and Zinc by Wavelength-Dispersive X-ray Fluorescence Spectroscopy
D 4929 Test Methods for Determination of Organic Chloride Content in Crude Oil
D 4951 Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry
D 5185 Test Method for Determination of Additive Elements, Wear Metals, and Contaminants in Used Lubricating Oils and Determination of Selected Elements in Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP–AES)
D 5480 Test Method for Motor Oil Volatility by Gas Chromatography[3]
E 1687 Test Method for Determining Carcinogenic Potential of Virgin Base Oils in Metalworking Fluids

2.2 *Government Standard:*
EPA8120, Chlorinated Hydrocarbons by GC/MS, EPA SW-846[4]

2.3 *Other Standards:*
IP 346, Polycyclic Aromatics and Other Species in Petroleum Fractions by Dimethyl Sulfoxide—Refractive Index Method[5]
CEC L-40-A-93 Evaporation Loss of Lubricating Oils (NOACK)[6]
JPI-5S-41-93, Method B, Determination of Evaporation Loss of Engine Oils (Unified NOACK)[7]
29 CFR Part 1910 Hazard Communication; Interpretation Regarding Lubricity Oils, Federal Register, Part 50 (245), pp. 5182–5185.[8]

3. Terminology

3.1 *Definitions*—For definition of standard terms used in this guide, see Terminology D 4175 or ASTM Dictionary of Engineering Science and Technology.

3.1.1 *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.

3.1.2 *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.

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

3.1.3.1 *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

[3] Withdrawn.

[4] 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.

[5] *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.

[6] Available from Commission of the European Communities, Rue De La Loi, B-1049B Rux Elles, Belgium.

[7] Available from Japanese Petroleum Institute, Keidanren Kaikan, 9-4 Ohtemachi 1-Chome, Chiyoda-ku, Tokyo.

[8] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

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.

5. Significance and Use

5.1 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 in some or all applications. A discussion of each of the suggested properties and potential contaminants is provided in Appendix X1, 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.

5.3 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 the specific application should serve as the basis for acceptability.

5.4 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.

6. Sampling

6.1 Sampling of base oils may be required as part of the buyer/seller arrangement. If a sampling program is required, sampling in accordance with Practice D 4057 or a suitable alternative may be employed.

7. Procedure

7.1 *Application of Guide*:

7.1.1 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.

7.1.2 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.

7.1.3 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 be negotiated between the base oil buyer and the seller.

7.1.4 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 E 1687 or IP 346, but the Chronic Animal Bioassay Analysis (that is, mouse skin painting assay) represents the definitive test for the determination of carcinogenicity hazard of base oils.

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

7.1.5 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.

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

7.2 *Properties and Potential Contaminants*:

7.2.1 The following tables contain suggested properties, potential contaminants, and commonly used test methods that one might want to include in a base oil evaluation.

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

7.2.3 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.[9]

7.2.4 A discussion of the significance of each property is provided in Appendix X1.

8. Keywords

8.1 base oil; base stock; hydrocarbon; lubricants; oil

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

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

Property	Test Method
Physical properties	
Appearance	[C]
Color	D 1500
Density at 15°C, kg/m^3	D 1298, D 4052
Flash point,° C	D 92
Kinematic viscosity at 40°C and 100°C, mm^2/s (cSt)	D 445
Pour point, °C	D 97
Viscosity index	D 2270
Volatility at 371°C, % off	D 2887, D 5480
% Evaporation loss	NOACK (CEC L-40-A-93 or JPI-5S-41-93)
Water separability (demulsibility), 30 min, mL	D 1401
Compositional properties	
Carbon residue, % mass	D 524, D 189, D 4530
Nitrogen, mg/kg	D 4629
Precipitation number	D 91
Saturates, wt %	D 2007
Sulfur, wt %	D 2622, D 4294, D 3120

[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 for a discussion of each property.
[C] Refer to X1.1.1 for a discussion of this property.

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

	Typical Levels	Test Method
Chemical properties		
Acid number, mg KOH/g	≤0.10	D 974, D 664
Base number, mg KOH/g	≤0.30	D 4739, D 2896
Total chlorine, mg/kg	≤50	D 4929
Copper corrosion, 3 h at 100°C	1	D 130
Elemental analysis, mg/kg:		
Mg, Na, Ba, Cu, B, Pb, Mn, Ni, Si		D 5185
Al, As, Cd, Ca, Fe, P, Zn, Cr, Sn,		(Also, D 4628, D 4927, D 4951
Total of all above elements	≤25	have limited applicability)
Glycol, mg/kg	≤5	D 4291
PCB content, mg/kg	≤2	D 4059
Total volatile organic halogens, mg/kg	≤5	EPA 8120
Water, mg/kg	≤150	D 1744
Toxicological properties[C,D,E]		
Mutagenicity index	pass	E 1687
DMSO extractables, wt %	pass	IP 346
Chronic animal bioassay analysis, number tumor-bearing animals/test group (%)	pass[F]	[G]

[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 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 E 1687 or IP 346. There presently are no published chronic skin-painting studies with re-refined base oils.
[D] Local legislative and regulatory requirements may also apply when selecting the tests to be run.
[E] For further information, see Appendix X2.
[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.
[G] Refer to X1.4.3 for discussion on this test method.