



Standard Guide for Characterizing Hydrocarbon Lubricant Base Oils¹

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INTRODUCTION

This guide is generated in response to a request from automobile manufacturers that ASTM Committee D-2 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 mm²/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:

- D 91 Test Method for Precipitation Number of Lubricating Oils²
- D 92 Test Method for Flash and Fire Points by Cleveland Open Cup²
- D 97 Test Method for Pour Point of Petroleum Products²
- D 130 Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test²
- D 189 Test Method for Conradson Carbon Residue of Petroleum Products²
- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)²
- D 524 Test Method for Ramsbottom Carbon Residue of Petroleum Products²
- D 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration²
- D 974 Test Method for Acid and Base Number by Color-Indicator Titration²
- D 1298 Practice for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method²
- D 1401 Test Method for Water Separability of Petroleum Oils and Synthetic Fluids²
- D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)²
- D 1744 Test Method for Water in Liquid Petroleum Products by Karl Fischer Reagent²

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² *Annual Book of ASTM Standards*, Vol 5.01.

- D 2007 Test Method for Characteristic Groups in Rubber Extender and Processing Oils and Other Petroleum-Derived Oils by the Clay-Gel Adsorption Chromatographic Method²
- D 2270 Practice for Calculating Viscosity Index from Kinematic Viscosity at 40 and 100°C²
- D 2622 Test Method for Sulfur in Petroleum Products by X-Ray Spectrometry²
- D 2887 Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography²
- D 2896 Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration²
- D 3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry²
- D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter³
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³
- D 4059 Test Method for Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography⁴
- D 4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants³
- D 4291 Test Method for Trace Ethylene Glycol in Used Engine Oil³
- D 4294 Test Method for Sulfur in Petroleum Products by Energy-Dispersive X-Ray Fluorescence Spectrometry³
- D 4530 Test Method for Determination of Carbon Residue (Micro Method)³
- D 4628 Test Method for Analysis of Barium, Calcium, Magnesium, and Zinc in Unused Lubricating Oils by Atomic Absorption Spectrometry³
- D 4629 Test Method for Trace Nitrogen in Liquid Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection³
- D 4739 Test Method for Base Number Determination by Potentiometric Titration³
- D 4927 Test Methods for Elemental Analysis of Lubricant and Additive Components—Barium, Calcium, Phosphorus, Sulfur, and Zinc by Wavelength-Dispersive X-ray Fluorescence Spectroscopy³
- D 4929 Test Methods for Determination of Organic Chloride Content in Crude Oil³
- D 4951 Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry³
- 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)³
- D 5480 Test Method for Motor Oil Volatility by Gas Chromatography⁵
- E 1687 Test Method for Determining Carcinogenic Poten-

tial of Virgin Base Oils in Metalworking Fluids⁶

2.2 Government Standard:

EPA8120, Chlorinated Hydrocarbons by GC/MS, EPA SW-846⁷

2.3 Other Standards:

IP 346, Polycyclic Aromatics and Other Species in Petroleum Fractions by Dimethyl Sulfoxide—Refractive Index Method⁸

CEC L-40-A-93 Evaporation Loss of Lubricating Oils (NOACK)⁹

JPI-5S-41-93, Method B, Determination of Evaporation Loss of Engine Oils (Unified NOACK)¹⁰

29 CFR Part 1910 Hazard Communication; Interpretation Regarding Lubricity Oils, Federal Register, Part 50 (245), pp. 5182–5185.¹¹

3. Terminology

3.1 *Definitions*—For definition of standard terms used in this guide, see Terminology D 4175 or Compilation of ASTM Standard Definitions.

3.1.1 *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.

3.1.2 *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 *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 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.

⁶ *Annual Book of ASTM Standards*, Vol 11.03.

⁷ 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.

⁸ Standard Methods for Analysis and Testing of Petroleum and Related Products, Vol 2. Available from Institute of Petroleum, London, 61 New Cavendish St., W.I., England.

⁹ Available from Commission of the European Communities, Rue De La Loi, B-1049B Rux Elles, Belgium.

¹⁰ Available from Japanese Petroleum Institute, Keidanren Kaikan, 9-4 Ohtemachi 1-Chome, Chiyoda-ku, Tokyo.

¹¹ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

³ *Annual Book of ASTM Standards*, Vol 5.02.

⁴ *Annual Book of ASTM Standards*, Vol 10.03.

⁵ *Annual Book of ASTM Standards*, Vol 5.03.

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 chromatogra-

phy (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.¹²

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

¹² Available from ASTM Headquarters, Request 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

^ASpecific 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.

^BSee Appendix X1 for a discussion of each property.

^CRefer 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 have limited applicability)
Total of all above elements	≤25	
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

^ASpecific 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.

^BSee Appendix X1 for discussion of each property.

^CChronic 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.

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

^EFor further information, see Appendix X2.

^FPassing 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.

^GRefer to X1.4.3 for discussion on this test method.

8. Keywords

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

APPENDIXES

(Nonmandatory Information)

X1. SIGNIFICANCE OF PROPERTIES OF LUBRICANT BASE OILS

X1.1 Physical Properties

X1.1.1 *Appearance*—Fully acceptable lubricant base oils are typically observed to be clear and bright. Simple visual inspection of lubricant base oils may indicate the absence or presence of undesirable contaminants (for example, water, haze wax, suspended materials, and so forth). If such contaminants are present, more definitive testing is recommended to assess their effect on other base oil or finished lubricant functional properties.

X1.1.2 *Color*—Determination of the color of lubricant base oils is used mainly for manufacturing control purposes and is an important characteristic since color is readily observed by the user of the product. In some cases, the color may serve as an indication of the degree of refinement of the lubricant base oil. When the color range of a particular product is known, a variation outside the established range may indicate possible contamination with another product. However, color is not

always a reliable guide to product quality and should not be used indiscriminately in product specifications.

X1.1.3 *Density*—Accurate determination of density of petroleum products is necessary for the conversion of measured volumes to volumes at the standard temperature of 15°C or 60°F using appropriate conversion tables. Accurate density determinations become critical with respect to transfer of custody.

X1.1.4 *Flash Point*—Safe operation of mechanical equipment requires an adequately high flash point. Flash point measures the tendency of the sample to form a flammable mixture with air under controlled laboratory conditions. Flash point can indicate the possible presence of highly volatile flammable materials in a relatively nonvolatile or nonflammable material, such as lubricant base oils.

X1.1.5 *Kinematic Viscosity*—Correct operation of equipment depends upon the appropriate viscosity of the liquid being

used. Accurate measurement of the kinematic viscosity of lubricant base oils is essential and fundamental in the formulation of lubricants, ensuring that product specifications and performance capabilities can be met.

X1.1.6 Pour Point—The pour point of a lubricant base oil is an indication of the lowest temperature of its utility for certain applications. The pour point is a function of the severity of the dewaxing operation employed in the refining process, and the chemical composition (normal paraffin content and carbon distribution) of the base oil.

X1.1.7 Viscosity Index—Viscosity index is an indicator of the variation in kinematic viscosity due to changes in temperature of a lubricant, indexed between 40°C and 100°C. A higher viscosity index indicates a smaller decrease in kinematic viscosity with increasing temperature of the lubricant.

X1.1.8 Volatility—Finished oil volatility is primarily a function of lubricant base oil volatility but can be influenced by the lubricant additives. Volatile organic compounds which evaporate from an engine crankcase may contribute to airborne hydrocarbon emissions as well as engine oil consumption.

X1.1.9 Water Separability (Demulsibility)—In service, lubricants are exposed to water contamination and turbulence, resulting in the formation of emulsions. The ability of the lubricant base oil to separate from water is critical to the successful formulation and performance of some lubricants. See X1.3.9, regarding water content, for related information.

X1.2 Compositional Properties

X1.2.1 Carbon Residue—Knowledge of the carbon residue, or coke- and ash-forming tendencies of lubricant base oils, provides an indication of suitability for high-temperature lubricant applications. In high-temperature applications, carbon residue can contribute to engine deposits and wear. High values of carbon residue indicate that a lubricant base oil may be unfit for this purpose.

X1.2.2 Nitrogen Content—Nitrogen is a naturally occurring element in crude petroleum and small quantities of nitrogen-containing compounds are frequently found in lubricant base oils. In addition, many lubricant additives contain nitrogen compounds. The concentration of nitrogen is often used as a measure of the presence of nitrogen-containing additives for quality purposes.

X1.2.3 Precipitation Number—The precipitation number is sometimes referred to in the industry as *asphaltenes*, since petroleum naphtha insolubles is the result reported. Low values of precipitation number are desirable because they provide an indication that potentially reactive hydrocarbon insoluble materials are not present in the lubricant base oil. The presence of hydrocarbon insoluble material provides an indication of incomplete base oil processing.

X1.2.4 Saturates Content—Chemical composition can have an effect on the characteristics and performance capabilities of lubricant base oils in concert with formulation additives. The saturates level is a function of crude oil source, refining sequence, and refining process severity.

X1.2.5 Sulfur Content—Sulfur is a naturally occurring element in crude petroleum and may act as an antioxidant in base oils. With respect to lubricant base oils, the sulfur content is a function of crude source, refining process, and processing

severity. Knowledge of the presence of sulfur-containing compounds in lubricant base oils may also be important in predicting the potential for corrosion. See X1.3.4, regarding copper corrosion, for related information.

X1.3 Chemical Properties

X1.3.1 Acid Number—A low acid number for the lubricant base oil portion of formulated lubricants is necessary to minimize the potential for metal corrosion and to maximize the life of the system being lubricated. High values for acid number of lubricant base oils provide an indication that oxidation reaction by-products may be present which should have been neutralized or removed in the re-refining process.

X1.3.2 Base Number—A low base number for the lubricant base oil portion of formulated lubricants is necessary to ensure that oxidation reaction degradation products have been effectively neutralized or removed in the re-refining process.

X1.3.3 Chlorine Content—This is an indirect indication of contamination. See X1.3.7 and X1.3.8, regarding PCB content and volatile organic halides, for related information.

X1.3.4 Copper Corrosion—Some sulfur compounds in lubricant base oils can have a corroding action on copper-containing metals and this corrosivity is not necessarily related directly to the total sulfur content. The effect can vary according to the chemical types of sulfur compounds present. The copper strip corrosion test is designed to assess the relative degree of corrosivity.

X1.3.5 Elemental Analysis—Lubricant base oils from a capable refining process are typically composed of carbon and hydrogen, with smaller concentrations of sulfur, nitrogen, and oxygen. Lubricant base oils should be essentially free of metallic elements. Sources of metallic elements potentially present in lubricant base oils include crude oil, refining or processing aids, residual lubricant additives, and residual corrosion or wear metals not removed in the re-refining process.

X1.3.6 Glycol Content—Engine oils can become contaminated with ethylene glycol from the engine coolant system during service. Used engine oils may be collected and re-refined into lubricant base oils. High glycol contents in lubricant base oils indicate a deficiency in the re-refining process to adequately remove this contaminant.

X1.3.7 PCB Content—Lubricant base oils must be free of PCB (polychlorinated biphenyl) compounds to be suitable for use in commerce. Historically, it has been demonstrated that PCBs are not present in lubricant base oils manufactured from virgin crude petroleum sources. However, used PCB containing fluids may inadvertently be admixed with used lubricating fluids destined for re-refining into lubricant base oils.

X1.3.8 Volatile Organic Halides—Lubricant base oils should be essentially free of volatile organic halides because capable refining and re-refining processes would effectively remove all traces of these materials. The presence of volatile organic halides in lubricant base oils indicates contamination with chlorinated solvents normally used in metalworking fluids and automotive parts washing fluids, or improper transportation of base oils. See X1.3.3, regarding chlorine content, for related information.

X1.3.9 Water Content— Knowledge of the water content of lubricant base oils may be important to adequately determine emulsibility or demulsibility characteristics of formulated lubricants (see X1.1.9 regarding water separability for related information) as well as concern for hydrolytic stability of additives and dielectric conductivity. Water is dissolved in all base oils at low concentrations when stored under atmospheric conditions in the presence of air. However, higher concentrations can occur when base oils are contaminated from external water sources.

X1.4 Toxicological Properties

X1.4.1 Mutagenicity Index (MI)—Test Method E 1687 describes a microbiological test procedure based upon a salmonella mutagenesis assay. It can be used as a screening technique to detect the dermal cancer hazard in lubricant base oils. Users should be well-versed in the conduct of the assay and conversant with the chemical and physical properties of petroleum products. The fundamental end point calculated from the data produced from this test method is the mutagenicity index (MI), which is used to predict the potential for base oils to elicit dermal carcinogenicity. Such a prediction should be evaluated on a case-by-case basis and may be more accurate when the MI and the results of an IP 346 test (see X1.4.2) are considered together. However, the standard mouse skin-painting bioassay is the most definitive test for such a prediction (see X1.4.3, regarding chronic animal bioassay analysis, for related information). Test Method E 1687 is not recommended as the sole testing procedure for base oils which have viscosities less than 18 cSt at 40°C (90 SUS at 100°F) nor are data available which correlate MI with skin-painting assays for re-refined base oils. **(1,2)**¹³

X1.4.2 Dimethyl Sulfoxide (DMSO) Extractables—This test concentrates and estimates polynuclear aromatic compounds (PAC), aromatic hydrocarbons, and related sulfur and nitrogen compounds, containing fused aromatic rings. These rings may have short alkyl or cyclo-alkyl groups as substituents. Polynuclear aromatics are compounds that may be normally found in crude petroleum and to a lesser extent in lubricant base oils; some of these materials have been shown to cause cancer. Minimum concentrations of these materials may be beneficial in formulated lubricants because they are believed to contribute

natural oxidation stability, seal swell, and enhanced additive solubility characteristics.

X1.4.2.1 The IP 346 method is a gravimetric procedure in which a sample of oil is diluted with cyclohexane and extracted twice with DMSO. The sample is cut so as to exclude material boiling below 300°C. The resulting extract includes such things as the 3 to 7-ring polycyclic aromatic hydrocarbons (some are known carcinogens) in the test sample but it is recognized that the method extracts other materials as well.

X1.4.2.2 The affinity of DMSO to specific hydrocarbon species increases as the number of aromatic rings increases, decreases as the length of the side chain increases, and is not selective between carcinogenic and noncarcinogenic PACs. This resulting DMSO extract is therefore larger, in terms of weight percent, than would be the case if the method extracted only carcinogens.

X1.4.2.3 Nevertheless, the percentage of DMSO extractables resulting from this test can be used to predict the potential for base oils to elicit dermal carcinogenicity and should be evaluated on a case-by-case basis. Such a prediction may be more accurate when the results are considered together with the results of Test Method E 1687 (see X1.4.1). However, the standard skin-painting bioassay is the most definitive test for such a prediction (see X1.4.3, regarding chronic animal bioassay analysis for related information) **(3)**.

X1.4.3 Chronic Animal Bioassay Analysis—This test, the mouse skin bioassay, is the animal test model of choice to predict/assess the carcinogenicity hazard of petroleum products. Mice are dosed dermally with 25 to 100 µL of test material two to three times per week for a total duration ranging from 18 months to the lifetime of the animal. The development and characterization of tumors provides evidence of carcinogenicity hazard of a petroleum product. The time of tumor development provides an indication of the carcinogenic potency of the material. Mouse skin bioassay is a sensitive indicator of polynuclear aromatic hydrocarbons, the carcinogenic moiety of lubricant base oils. Passing results in this test 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 animals. Analysis of the data should be performed on a case-by-case basis using sound scientific judgment and appropriate statistical analyses **(4,5)**.

¹³ The boldface numbers refer to the list of references at the end of this standard.

X2. REGULATORY AND LEGISLATIVE CLASSIFICATION OF BASE STOCKS AND BASE OILS

X2.1 Carcinogenicity Classification of Base Stocks/Oils

X2.1.1 In various regions of the world there is existing or pending legislation covering the classification of hydrocarbon base stocks and base oils for carcinogenic hazard that may need to be considered in addition to, or in place of, some of the parameters provided in this guide.

X2.1.2 United States— In the United States, the carcinogenicity classification of virgin base stocks is covered by 29 CFR Part 1910. For mineral oils, OSHA base their classification on

the results of the IARC (International Agency for Research on Cancer) review of the available carcinogenicity data on base stocks **(6,7)**. Under the OSHA rules, base stocks are classified on the basis of their processing history. Thus, *mildly* hydrotreated or *mildly* solvent refined base stocks are considered to possess possible carcinogenic potential, whereas *severely* hydrotreated or *severely* solvent refined base stocks are not considered to present a carcinogenic hazard. OSHA has from time to time given interpretative guidance on the determination

of processing severity (8) (see 29 CFR Part 1910). OSHA rule making clearly requires chemical manufacturers and importers to evaluate the hazards of the chemicals they produce or import (29 CFR Part 1910).

X2.1.3 Europe—In Europe, the carcinogenicity classification of virgin base stocks is covered by an “Adaptation to Technical Progress of the Dangerous Substances Directive” (9). This states “that the classification (of a base stock) as a carcinogen need not apply if it can be shown that there is less than 3 % DMSO extractable compounds as measured by IP 346.” IP 346 is a standard Institute of Petroleum analytical method. The classification criteria established by the EU were based on a published correlation between the results of long term mouse skin painting studies on base stocks and the amount of their DMSO extract (3).

X2.1.4 Other—The situation in the rest of the world is less clear as regulatory frameworks are still developing. There are, however, indications that the US (OSHA) and European (EU)

approaches to the carcinogenicity classification of base stocks are favored by regulatory authorities in other regions of the world. The situation is somewhat mixed as some regulators may express a preference for one approach or another, whereas others may accept either criteria for classification.

X2.2 Re-refined oil—As yet, the issue of carcinogenicity classification of re-refined base stocks has not been considered by regulators in the US or the EU. There are some national technical specifications/guidelines for re-refined base stocks, but the parameters set out in these do not address health concerns or classification for possible carcinogenic potential (10,11). Re-refined oils is a generic term used to describe used oils that have been subjected to undefined types and severities of treatment/processing, producing products of different composition. Thus, no general rules can be laid down as to their carcinogenic properties (12). At present, they are classified and labelled for carcinogenicity on a case by case basis, which may be feedstock, site, and process specific.

X3. ADDITIONAL INFORMATION ON TESTING OF BASE STOCKS AND BASE OILS FOR TOXICOLOGICAL PROPERTIES

X3.1 For those interested in a more detailed discussion of the current significance given to each of the toxicological tests identified in the guide, comments are provided on the use and scope of the three toxicological properties for determining the carcinogenic potential or classification of base stocks and base oils.

X3.1.1 Long Term Mouse Skin Painting Bioassay—This is generally considered by regulatory authorities to be the definitive test for determining the dermal carcinogenic potential of a material. Notwithstanding this, regulators recognize that the test is expensive to perform, requires a large number of animals, and takes a long time to obtain results. The oil industry has sought to develop alternative strategies for predicting the carcinogenicity of base stocks (13,14). While it is recognized that there are no formal regulatory guidelines on the conduct and interpretation of skin painting studies, the general principles recommended in these respects by OECD, US NTP and Horton, et. al. are usually followed (15, 16, 17).

X3.1.2 Dimethyl Sulphoxide (DMSO) Extractables (IP346)—IP 346 is a standard analytical method developed by the Institute of Petroleum. Based on a correlation with long term skin painting data, the percentage mass of DMSO extractables can be used to predict the potential for virgin base stocks to elicit dermal carcinogenicity; there is no published correlation with re-refined base stocks/oils (18). Under EU legislation, it is stated that the classification of a base stock as carcinogenic need not apply if it can be shown that the substance contains less than 3 % DMSO extract as measured by IP 346 (9). In the EU, IP 346 is the sole regulatory marker for the purposes of carcinogenicity classification of virgin base stocks and other data, such as results of long term skin painting tests or mutagenicity studies, cannot be used to override classification (9).

X3.1.2.1 The scope and limitations of the IP 346 method for carcinogenicity classification are defined by the IP methodology, the CONCAWE review, and the EU Dangerous Substances Directive (9,18). Briefly, the published scope and limitations are:

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

(b) As the EU legislation is framed presently, the IP 346 marker only applies to individual virgin base stocks and cannot be used for classification of mixtures of base stocks, or products containing additives

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

X3.1.3 Mutagenicity Index (MI) — Test Method E 1687 (Modified Ames)—This is a short-term microbiological assay that has been developed to detect the presence of mutagens in lubricant base stocks. A correlation of mutagenicity with carcinogenicity was developed with results from this assay and those of long term skin painting data on virgin mineral oils; there is no published correlation with re-refined base stocks/oils (19, 20, 21). Guidance on protocols, interpretation and pass/fail criteria for modified Ames tests are included in the above referenced papers.

X3.1.3.1 The scope and limitations of the Modified Ames test are defined in Test Method E 1687. Briefly the published scope and limitations are:

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

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

(c) The correlation of mutagenicity and carcinogenicity with PNA content is restricted to oils distilling in the range from approximately 250°C (that is, viscosities >18 cSt at 40°C) up to, but not including, vacuum residua or materials produced from them (boiling points >550°C).

REFERENCES

- (1) Blackburn, G. R., Deitch, R. A., Schreiner, C. A., Mehlman, M. A., and Mackerer, C. R., "Estimation of Dermal Carcinogenic Activity of Petroleum Fractions Using a Modified Ames Assay," *Cell Biology and Toxicology*, Vol 1(1), 1984, pp. 40–48.
- (2) Skisak, C. M., Venier, C. M., and Baker, D. O., "Ames Tests of Lubricating Oil Products: The Mutagenic Potency Index," *In Vitro Toxicology*, Vol 1, 1987, pp. 263–276.
- (3) CONCAWE Report No. 94/51, "The Use of the Dimethylsulfoxide (DMSO) Extract by the IP346 Method as an Indicator of the Carcinogenicity of Lubricant Base Oils and Distillate Aromatic Extracts," The Hague, Netherlands, February 1994.
- (4) McKee, R. H., Lewis, S. C., and Egan, G. F., "Experience Gained by the Petroleum Industry in the Conduct of Dermal Carcinogenesis Bioassays," *Skin Carcinogenesis: Mechanisms and Human Relevance*, T. Slaga, A. Klein, R. Boutwell, D. Stephenson, H. Spitzer, B. D'Motto, and Alan R. Liss, eds, New York, 1989, pp. 363–379.
- (5) Chasey, K. L., and McKee, R. H., "Evaluation of the Dermal Carcinogenicity of Lubricant Base Oils by the Mouse Skin Painting Bioassay and Other Proposed Methods," *Journal of Applied Toxicology*, Vol 13(1), 1993, pp. 57–65.
- (6) IARC Monograph on the Evaluation of the Carcinogenic Risk to Humans, Vol 33. Polynuclear Aromatic Compounds, Part 2. Carbon Blacks, Mineral Oils and Some Nitroarenes. Lyon: International Agency for Research on Cancer, 1984, pp. 87–168.
- (7) IARC Monograph on the Evaluation of the Carcinogenic Risk to Humans, Supplement 7, Overall Evaluation of Carcinogenicity: an Updating of IARC Monographs Volumes 1 to 42. Lyon: International Agency for Research on Cancer, 1987, pp. 252–254.
- (8) OSHA Interpretation; Solvent Refined Lubricating Oils. Letter to San Joaquin Refining Co., Personal Communication, 1986.
- (9) Commission Directive 94/69/EC of 19 December 1994 Adapting to Technical Progress for the Twenty-first Time Council Directive 67/548/EEC on the Approximation of Laws, Regulations and Administrative Provisions Relating to the Classification, Packaging and Labelling of Dangerous Substances. Official Journal of the European Communities No. L381, European Commission, 1994.
- (10) Requirements Specification EC-03-92, Environmental Choice New Zealand. Oil: Re-refined Lubricating Oil. Telarc, 1992.
- (11) ECP-01-89, Environment Canada. Environmental Choice Program. Automotive Engine Oil 1996.
- (12) CONCAWE Report No. 95/59, "The Classification and Labelling of Petroleum Substances According to the EU Dangerous Substances Directive," 1995.
- (13) "Investigations into the Relationship Between the Composition of Mineral Oils and Their Ability to Cause Skin Cancer In Mice." CONCAWE Report No. 4/85, 1985.
- (14) "The Use Of Short-term Tests to Predict the Carcinogenicity of Mineral Oils." CONCAWE Report No. 88/57, 1988.
- (15) "Guideline for Testing of Chemicals" OECD No 451, Adopted May 12, 1981.
- (16) "Explanation of Levels of Evidence of Carcinogenic Activity," US NTP Adopted June 1983 revised March 1986.
- (17) Horton, A., et al, "Carcinogenesis of the Skin: A Re-examination of Methods for the Quantitative Measurement of the Potencies of Complex Materials," *Cancer Research* Vol. 15 1955, pp. 701–709.
- (18) "The Use of the Dimethyl Sulphoxide (DMSO) Extract by the IP 346 Method as an Indicator of the Carcinogenicity of Lubricant Base Oils and Distillate Aromatic Extracts," CONCAWE Report No. 94/51, 1994.
- (19) Blackburn G.R., Deitch R.A., Schreiner C.A., et al "Estimation of the Dermal Carcinogenic Activity of Petroleum Fractions using a Modified Ames Assay," *Cell Biology and Toxicology* Vol 1 (1), 1984, pp. 67–80.
- (20) Blackburn G.R., Deitch R.A., Schreiner C.A., et al, "Predicting Carcinogenicity of Petroleum Distillation Fractions Using a Modified Salmonella Mutagenicity Assay," *Cell Biology and Toxicology* Vol 2 (1), 1986, pp. 63–84.
- (21) Roy T.A., Johnson S.W., Blackburn G.R., et al, "Correlation of Mutagenic and Dermal Carcinogenic Activities of Mineral Oils with Polycyclic Aromatic Compound Content," *Fundamental Applied Toxicology*, Vol 10, 1988, pp. 466–476.

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