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(R) PHYSICAL AND CHEMICAL PROPERTIES OF ENGINE OILS

Foreword—This document discusses a number of the physical and chemical properties of new and used engine oils. Where appropriate, standardized methods of test for these properties are indicated and a detailed listing included in the references section. This document provides those concerned with the design and maintenance of internal combustion engines with information relative to the terms used to describe engine lubricants.

This document may be used as a general guide to engine oil properties and as an outline for establishing oil quality inspection and maintenance programs.

1. Scope

1. Scope—This SAE Information Report reviews the various physical and chemical properties of engine oils and provides references to test methods and standards used to measure these properties. It also includes general references on the subject of engine oils, base stocks, and additives.

2. References

2.1 Applicable Documents—The following publications form a part of this specification to the extent specified herein. The latest issue of SAE, ASTM, API, and CEC publications shall apply.

2.1.1 SAE PUBLICATIONS—Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

SAE J183—Engine Oil Performance and Engine Service Classification (Other than "Energy Conserving")
SAE J300—Engine Oil Viscosity Classification
SAE J304—Engine Oil Tests
SAE J1423—Classification of Energy-Conserving Engine Oil for Passenger Cars, Vans, and Light Duty Trucks
SAE J2227—International Tests and Specifications for Automotive Engine Oils

2.1.2 ASTM PUBLICATIONS—Available from ASTM, 1916 Race Street, Philadelphia, PA 19103-1187.

ASTM DS 39b—Viscosity Index Tables for Celsius Temperatures
ASTM D 56—Test Method for Flash Point by Tag Closed Tester
ASTM D 91—Test Method for Precipitation Number of Lubricating Oils
ASTM D 92—Test Method for Flash and Fire Points by Cleveland Open Cup
ASTM D 93—Test Methods for Flash Point by Pensky-Martens Closed Tester
ASTM D 95—Test Method for Water in Petroleum Products and Bituminous Materials by Distillation
ASTM D 97—Test Methods for Pour Point of Petroleum Oils
ASTM D 156—Test Method for Saybolt Color of Petroleum Products (Saybolt Chromometer Method)

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ASTM D 189—Test Method for Conradson Carbon Residue of Petroleum Products
ASTM D 287—Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)
ASTM D 322—Test Method for Gasoline Diluent in Used Gasoline Engine Oils by Distillation
ASTM D 341—Viscosity-Temperature Charts for Liquid Petroleum Products
ASTM D 445—Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)
ASTM D 482—Test Method for Ash from Petroleum Products
ASTM D 524—Test Method for Ramsbottom Carbon Residue of Petroleum Products
ASTM D 664—Test Method for Acid Number by Potentiometric Titration
ASTM D 874—Test Method for Sulfated Ash from Lubricating Oils and Additives
ASTM D 892—Test Method for Foaming Characteristics of Lubricating Oils
ASTM D 893—Test Method for Insolubles in Used Lubricating Oils
ASTM D 974—Test Method for Acid and Base Number by Color Indicator Titration
ASTM D 1160—Method for Distillation of Petroleum Products at Reduced Pressure
ASTM D 1298—Test Method for Density, Relative Density (Specific Gravity) or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
ASTM D 1310—Test Method for Flash Point and Fire Points of Liquids by Tag Open Cup Apparatus
ASTM D 1500—Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
ASTM D 2270—Method for Calculating Viscosity Index from Kinematic Viscosity at 40 and 100 °C
ASTM D 2500—Standard Test Method for Cloud Point of Petroleum Oils
ASTM D 2887—Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography
ASTM D 2896—Test Method for Total Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration
ASTM D 2982—Test Method for Detecting Glycol-Base Antifreeze in used Lubricating Oils
ASTM D 3244—Standard Practice for Utilization of Test Data to Determine Conformance with Specifications
ASTM D 3524—Test Method for Diesel Fuel Diluent in Used Diesel Engine Oil by Gas Chromatography
ASTM D 3525—Test Method for Gasoline Diluent in Used Gasoline Engine Oils by Gas Chromatography
ASTM D 3607—Method for Removing Volatile Contaminants from Used Engine Oils by Stripping
ASTM D 3828—Test Method for Flash Point by Setaflash Closed Tester
ASTM D 3829—Test Method for Predicting the Borderline Pumping Temperature of Engine Oil
ASTM D 3945—Standard Test Methods for Shear Stability of Polymer-Containing Fluids Using a Diesel Injector Nozzle
ASTM D 4055—Test Method for Pentane Insolubles by Membrane Filtration
ASTM D 4485—Performance Specification for Automotive Engine Oils
ASTM D 4530—Test Method for Micro-Carbon Residue of Petroleum Products
ASTM D 4628—Test Method for Analysis of Barium, Calcium, Magnesium, and Zinc in Unused Lubricating Oils by Atomic Absorption Spectrometry
ASTM D 4683—Test Method for Measuring Viscosity at High Temperature and High Shear Rate by Tapered Bearing Simulator
ASTM D 4684—Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature
ASTM D 4739—Test Method for Base Number Determination by Potentiometric Titration
ASTM D 4741—Test Method for the Measurement of Viscosity at High Temperature and High Shear Rate by Tapered Plug Viscometer
ASTM 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
ASTM D 4951—Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry

ASTM D 5002—Standard Test Method for Density and Relative Density of Crude Oil by Digital Density Analyzer
ASTM D 5119—Test Method for Evaluation of Automotive Engine Oils in the CRC L-38 Spark Ignition Engine
ASTM D 5133—Standard Test Method for Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature Scanning Technique
ASTM D 5185—Standard Test Method for Determination of Additive Elements, Wear Metals, and Contaminants in Used Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry
ASTM D 5293—Standard Test Method for Apparent Viscosity of Engine Oils Between -5 °C and -30 °C Using the Cold Cranking Simulator
ASTM E 1131—Test Method for Compositional Analysis by Thermogravimetry
ASTM MNL 1—Manual on the Significance of Tests for Petroleum Products: 5th Edition
ASTM STP 1068—High Temperature/High Shear (HTHS) Oil Viscosity: Measurement and Relation to Engine Operation
ASTM STP 1143—Low Temperature Lubricant Rheology Measurement Relevance to Engine Operations

2.1.3 OTHER PUBLICATIONS

API Publication 1509, latest edition, plus revisions
Federal Test Method Standard No. 791C, Method 203.1, "Pour Stability of Lubricating Oils"
Federal Test Method Standard No. 791C, Method 3470.1, "Homogeneity and Miscibility of Oils"
American Society of Lubrication Engineers, 1951, "The Physical Properties of Lubricants"
General Motors Engineering Standard GM 9099P, "Engine Oil Filterability Test (EOFT)," May 1980
CEC L-40-T-87, "Evaporative Loss," NOACK (DIN 51581) Method
CEC L-14-A-88 "Evaluation of the Mechanical Shear Stability of Lubricating Oils Containing Polymers" Method
C.M. Georgi, "Motor Oils and Engine Lubrication," New York: Reinhold Publishing Corporation, 1950
William A. Gruse, "Motor Oils, Performance and Evaluation," New York: Reinhold Publishing Corporation, 1967
A. Schilling, "Automotive Engine Lubrication," Broseley, England: Scientific Publications (G.B.) Ltd., 1972
R.C. Gunderson and A.W. Hart, "Synthetic Lubricants," New York: Reinhold Publishing Corporation, 1962
Dieter Klamann, "Lubricants and Related Products," Weinheim: Verlag Chemie (F.R.G.) GmbH, 1984
M. Campen, D. Kendrick, and A. Markin, "Growing Use of Synlubes," Hydrocarbon Processing, February 1982
G.J. Schilling and G.S. Bright, "Fuel and Lubricant Additives—II. Lubricant Additives," Lubrication Vol 63, No. 2, 1977
N. Benfaremo and C.S. Liu, "Crankcase Engine Oil Additives," Lubrication Vol 76, No. 1, 1990

3. *General Description of Engine Oil Components*—Modern engine oils consist of (1) base stocks and (2) the additives that are necessary to produce the required finished product performance. These engine oil components will be described in the following sections.

3.1 Base Stocks—Base stock properties have assumed increased importance with the addition of Base Oil Interchangeability Guidelines to the API Engine Oil Licensing and Certification System. These guidelines are intended to determine engine sequence tests required for variations in base stock compositions. See API Publication 1509.

3.1.1 REFINED PETROLEUM BASE STOCKS—Crude petroleum oil as it comes from the ground is a mixture of literally hundreds of hydrocarbon molecules of three basic types—paraffinic, naphthenic, and aromatic. Crude oils are classified according to the predominant type of hydrocarbon molecules they contain.

The first step in refining crude oil into useful products is the separation according to boiling range by atmospheric and/or vacuum distillation. The various fractions are then further processed into gaseous products, gasoline, diesel and burner fuels, lubricating oil stock, asphalt, etc. The lubricating oil stock is vacuum distilled, providing a series of base stocks of various levels of volatility and viscosity. The less viscous distillate fractions are called “neutrals” and the higher viscosity residual fractions are called “bright stocks.” These fractions generally require further treatment to make them suitable for use as engine oils.

Historically, a nomenclature has evolved to identify neutral and bright stock fractions by their Saybolt viscosities. Neutral fractions are referred to by their nominal viscosity at 100 °F in Saybolt Universal Seconds (SUS). For example, a 150 neutral is a distillate fraction with a nominal viscosity of 29 mm^2/s (cSt) at 40 °C (150 SUS at 100 °F). Generally speaking, the viscosity for the distillate (neutral) fractions range from about 11 to 150 mm^2/s (cSt) at 40 °C (60 to 700 SUS).

In similar fashion, bright stocks are referred to by their nominal SUS viscosity at 210 °F rather than 100 °F. For example, a 150 bright stock is a residual fraction with a nominal viscosity of 30.6 mm^2/s (cSt) at 100 °C (150 SUS at 210 °F). For comparison purposes, if the viscosity of the bright stocks were measured at 40 °C rather than 100 °C, they would range from 140 to about 1600 mm^2/s (cSt) (650 to 7400 SUS).

The as-distilled base stock fractions may contain nitrogen- and sulfur-containing compounds, metal-containing compounds, and aromatic hydrocarbons of various structures. Many of these compounds can adversely affect the stability and performance properties of base stocks and the ability of various additives to enhance these properties for engine oil applications. These compounds are usually removed through extraction processes, using solvents such as phenol, furfural, or N-methyl pyrrolidone, or are modified by hydrotreating or hydrocracking.

Both hydrotreated and hydrocracked base stocks are typically composed of higher percentages of saturates and reduced sulfur contents relative to solvent refined petroleum base stocks. Hydrocracking can also increase the proportion of iso-paraffins at the expense of the less desirable hydrocarbons.

Waxy materials present in the base stock fractions may crystallize and agglomerate or congeal at low temperatures and thereby impede low temperature flow. These materials may be removed by solvent dewaxing processes employing solvents such as methylethyl ketone or propane, or by catalytic dewaxing.

These extraction and modification processes can be carried out either before or after final distillation into viscosity fractions. The choice depends on the processes employed.

The physical and chemical properties of the finished base stocks (often referred to as “virgin”) will not be solely a function of crude source, but also will be dependent on the processes employed and the extent of refining employed.

3.1.2 RE-REFINED OR RECYCLED PETROLEUM BASE STOCKS—Used lubricating oils have been involved in recycling processes for over 65 years. Recycling normally involves the removal of volatile components produced in use as well as water, insolubles, and dirt. Little, if any, additional processing is involved. The resulting oil is not normally considered to be suitable for use in modern engines. However, it is often blended with other materials and burned as a fuel.

Recently, legislation and technical improvements in re-refining processes have increased the interest in re-refined oils. Re-refined petroleum base stocks may be manufactured from used oil by re-refining processes. Re-refined stock shall be substantially free from additives and from contaminants introduced from the re-refining process or from previous use. Re-refined oils can undergo one or more of the following processes: water separation, additive separation, solvent extraction, hydrotreating, and re-fractionation. The resulting finished, re-refined oil is often virtually indistinguishable from good quality virgin base stocks. These re-refined oils may be suitable for use in modern engines when treated with appropriate additives.

3.1.3 SYNTHETIC BASE STOCKS—Certain chemical compounds have been found to be suitable as base stocks for engine oil. These are referred to as synthetic lubricants and are defined as having been produced by chemical synthesis. These are manufactured by organic reactions such as alkylation, condensation, esterification, polymerization, etc. Starting materials may be one or more relatively pure organic compounds. Generally of simple composition, these compounds are obtained by chemically processing fractions from petroleum, natural gas, vegetable, or animal oil components. When vegetable or animal oil base lubricants are derived from natural, nonpetroleum sources rather than from synthesis, they are not considered synthetic lubricants unless the naturally occurring product has been chemically changed.

Classes of chemical compounds that might be used as synthetic base stocks after processing are shown in Table 1 along with distinct generic identification of the resulting fluids. A synthetic lubricant base stock may consist of any of the fluids shown in Table 1, or a mixture of compatible base fluids. This blending is usually practiced to enhance physical properties.

TABLE 1—CLASSIFICATION OF SYNTHETIC BASE FLUIDS

Class	Synthetic Fluids (Examples)
Synthetic Hydrocarbons	
Alkylated Aromatics	Alkylbenzenes
Polyolefins	Polyalphaolefins (Hydrogenated) Polybutenes
Organic Esters	
Dibasic Acid Esters (Diesters)	Adipates, Azelates, Dodecanedioates
Polyol Esters	Neopentyl or Hindered Esters
Polyesters	Dimer Acid Esters
Others	
Halogenated Hydrocarbons	Chlorofluorocarbon Polymers Fluoroesters, Fluoroethers
Phosphate Esters	Phosphate Esters of Isopropyl Phenol and Cresylic Acids
Polyglycols	Polyalkylene Glycols
Polyphenyl Esters	Meta bis (m-Phenoxyphenyl) benzene
Silicate Esters	Disiloxane Derivatives
Silicones	Phenyl, Methyl, and Alkylmethyl Silicones

Some synthetic base stocks are compatible with petroleum base stocks, and the two types may be blended to obtain desired physical and chemical properties. Such combinations are referred to as "partial synthetic base stocks."

Some synthetic base stocks are not compatible with either other synthetics or with petroleum base stocks. Therefore, lubricants containing synthetic base stocks should not be indiscriminately mixed.

The additive agents necessary in petroleum base stocks, synthetic base stocks, or partial synthetic blends intended for engine oils are also synthesized materials. However, even though these materials are synthesized, they should be referred to as additives and not included in the base stock description.

3.2 **Additive Agents**—A lubricant additive agent is defined as a material designed to enhance the performance properties of the base stock or to impart to the base stock properties that do not naturally exist with the base stock. These additive agents are used at concentration levels ranging from several parts per million to greater than 10 volume percent. Generally, additives are materials that have been chemically synthesized to provide the desired performance features, and they frequently contain an oil-solubilizing hydrocarbon portion as part of the molecule. Some additive agents are naturally occurring materials that have undergone only minor modifications to obtain the desired property. Additives can carry out their task of enhancing or imparting new properties to an oil in one of three ways—protection of engine surfaces, modification of oil properties, protection of base stocks. Engine protectors include seal swell agents, antiwear agents, extreme pressure (EP) agents, antirust agents, corrosion inhibitors, detergents, dispersants, and friction modifiers. Oil modifiers include pour point depressants, antifoam agents, and viscosity index (VI) improvers. Base stock protectors include antioxidants and metal deactivators. Some additives possess multifunctional properties.

Additive combinations contribute performance features which are required to satisfy the lubrication needs of modern engines under the most severe conditions and currently recommended oil change intervals. If additives have been put into the base stock to increase its commercial value to those who will use it for formulating engine oil, care should be taken to identify such additives so that further additive treatments will be compatible.

4. ***Physical and Chemical Properties***—Understanding and agreeing on the methods of measurement of the physical and chemical properties of base oils and formulated engine oils can assist the user, the oil refiner, and the formulator to define a consistently uniform product. These properties are often used to establish acceptable levels of additive components in finished oils. Although oil performance in the engine is related to base stock and additive composition, it is often difficult to assign a specific aspect of such performance totally to the use of a specific additive or base stock. Part of the reason is that some of the physical and chemical properties of the oil overlap in their influences on engine performance and durability and it is presently difficult to directly and unambiguously attribute such effects to either the chemical or the physical properties of the oil. Progress in developing this level of understanding is being made. Some of these performance characteristics of engine oils are discussed in SAE J183, SAE J300, SAE J1423, and SAE J2227. At the present time, oil physical and chemical properties can be related to engine performance and durability only with the guidance of engine manufacturers and with appropriate and jointly accepted engine and/or field tests successfully completed on that oil.

At low temperatures where cranking/starting and engine oil pumpability are matters of concern, the physical properties of the engine oil can be directly related to its effects on any particular engine. Engine cranking and starting and oil pumpability are also related to a variety of other factors including engine response to oil rheology, as well as to nonrheological factors such as battery power and fuel volatility.

While the physical and chemical properties of an oil at operating temperatures are not related to oil performance in a simple way, these individual properties are meaningful and are related to the oil's ability to fulfill its function as a lubricant. These and the low temperature properties will be discussed in the following sections.

4.1 Viscosity—Viscosity of the engine oil is one of its most important and most evident properties. If sufficiently high, it is the source of the phenomenon of hydrodynamic lubrication in which the viscosity of the oil forces the bearing surface to ride on a thin film of oil and, thus, protect the lubricated surface from wear. Chemical additives, fuel dilution, contaminants from within and outside of the engine, wax in the oil, oil oxidation, volatilization, and many other materials found in or added to the oil affect the viscosity in advantageous or disadvantageous ways.

4.1.1 DEFINITION—Viscosity is defined as the internal resistance to flow of any fluid. It is expressed as follows in Equation 1:

$$\text{Dynamic Viscosity} = \frac{\text{Force / Sheared Area}}{\text{Velocity / Film Thickness}} = \frac{\text{Shear Stress}}{\text{Shear Rate}} \qquad \text{(Eq.1)}$$

The unit of measure for dynamic viscosity is the millipascal second (mPa·s), although the centipoise (cP) is also commonly used. One mPa·s equals 1 cP. Oils that exhibit a constant viscosity at all shear rates in this equation are known as "Newtonian" oils. In the absence of polymeric additives, most single grade oils are in this category at temperatures above their cloud point.

Oils that exhibit a viscosity which varies with changing shear rates in this equation are known as "non-Newtonian" oils. Multiviscosity graded oils formulated with polymeric additives are generally in this category.

Another form of viscometric expression involves the use of kinematic viscometers in which the liquid is driven by its own hydrodynamic head. This head varies directly with the density of the oil at the temperature of measurement. The relationship between kinematic and dynamic viscosity is as follows in Equation 2:

$$\text{Kinematic Viscosity} = \text{Dynamic Viscosity/Density of Liquid} \qquad \text{(Eq.2)}$$

The unit of measurement for kinematic viscosity is the millimeter squared per second (mm^2/s), although the centistoke (cSt) is commonly used. One mm^2/s equals 1 cSt. Density effects should be eliminated either by measuring the dynamic viscosity or by measuring kinematic viscosity and density at the temperature of interest and converting the values to dynamic viscosity.

4.1.2 VISCOSITY INDEX (VI)—Viscosity decreases rapidly with increasing temperature. For most oils, the relationship between viscosity and temperature can be approximated by the following empirical relationship in Equation 3:

$$\text{loglog(kinematic viscosity} + 0.7) = A + B\ \text{log(absolute temperature)} \qquad \text{(Eq.3)}$$

where:

A and B are constants, specific for each oil

This relationship, which is an approximation of the MacCoull, Walther, Wright equation, forms the basis for special viscosity temperature charts published in ASTM D 341. These charts permit the plotting of viscosity-temperature data as straight lines over the temperature range in which the oils are homogeneous liquids. The slope of these lines is a measure of the change in viscosity with temperature. It is dependent on the chemical composition of the oil and is described by an empirical relationship called VI. The higher the VI, the smaller the change in viscosity with temperature (slope). ASTM D 2270 is used to determine VI values. ASTM DS 39b is based on ASTM D 2270 and allows for more convenient determination of VI.

For engine oils, a relatively smaller change in viscosity with temperature (high VI) is desirable to provide a wider range of operating temperatures over which a given oil will provide satisfactory lubrication. At low temperatures, a relatively low viscosity oil is desirable to permit adequate cranking speed during starting, and then adequate flow to the oil pump and the entire engine oiling system after starting.

At high temperatures in a running engine, the oil viscosity must be high enough to maintain adequate film thickness between rotating or rubbing parts to minimize wear. Using a higher viscosity oil generally reduces oil consumption (past piston rings and valve guides) and blowby, but increases friction associated with oil film shearing in the piston/piston ring cylinder wall interface and bearings.

4.1.3 VISCOSITY INDEX IMPROVERS—To extend the upper temperature limit at which an oil will still provide satisfactory lubrication, polymeric additives, called Viscosity Index (VI) improvers, are widely used. Engine oils properly formulated with VI improvers generally contain lower viscosity base stocks which provide better low temperature cranking/starting and pumpability properties. As the oil temperature increases, the viscosity of the oil containing a VI improver decreases more slowly than the same oil without a VI improver, thus increasing the VI. The result is an oil that can give good starting/pumping response and is also effective in providing a more viscous oil film at operating temperatures than could be obtained with a single grade oil providing equivalent startability at low temperatures.

Oils containing a polymeric VI improver exhibit a decrease in viscosity as the shear rate or stress is increased. Because the viscosity of such oils depends on shear stress, they are called "non-Newtonian oils." Such change generally lasts only as long as the oil is operated under such high shear stress. When the shear stress is relieved, the oil reverts to its previous viscosity. This reversible decrease in viscosity due to shear is called "temporary shear (or viscosity) loss." When certain critically high shear stresses are imposed on a VI improver in oil solution, the viscosity contribution of the VI improver to both low and high shear rate viscosity can be permanently reduced. This nonreversible reduction in viscosity is called "permanent shear (or viscosity) loss." The magnitude of these temporary and permanent losses is dependent on the type and molecular weight of the VI improver used, as well as the actual service conditions.

The permanent shear stability characteristics of engine oils are evaluated by comparing the stripped viscosity of an engine oil after 10 h in the CRC L-38 engine test to the new oil viscosity. Shear stability can also be measured using CEC L-14-A-88.

4.1.4 VISCOSITY MEASUREMENT—The SAE J300 standard classifies oils into grades according to their kinematic viscosities measured at low shear rates and high temperature (100 °C), and their viscosities at high shear rate and high temperature (150 °C), and at both low and high shear rates at low temperatures (–5 to –35 °C).

Low shear rate kinematic viscosity is measured using ASTM D 445 and is reported in millimeters squared per second (mm^2/s), although the centistoke (cSt) is also commonly used. Kinematic viscosity is measured most commonly at 100 °C, and also at 40 °C if VI is to be determined.

At low temperature, the high shear rate viscosity is measured by ASTM D 5293. This is a multitemperature cold cranking simulator method. The low temperature, low shear rate viscosity is measured by ASTM D 4684. Both ASTM D 5293 and ASTM D 4684 report viscosities in millipascal seconds (mPa·s), although the centipoise (cP) is also commonly used. Results of both tests have been shown to correlate with engine starting and engine oil pumpability at low temperatures, although the precise correlation to modern engines has been questioned and is under active investigation with ASTM.

Oil viscosity at very high shear rates/stresses and at high temperatures (150 °C) is measured using ASTM 4683 or ASTM 4741 and is reported in millipascal seconds (mPa·s), although the centipoise (cP) is also commonly used. A comparable Capillary Viscometer method is currently being developed by ASTM. These methods are intended primarily to simulate operating conditions occurring in engine bearings.

4.2 Other Tests Pertinent to New and Used Oils

4.2.1 CLOUD POINT AND POUR POINT—The cloud point of a moisture-free oil is defined as the temperature at which a cloud or haze appears in the lower portion of the test oil when tested (i.e., cooled) by ASTM D 2500. The haze indicates the presence of some insoluble fractions, such as wax, at the temperature noted. In most applications, this haze will have little practical significance.

The pour point of an oil is defined as the lowest temperature at which the oil can be poured when tested by ASTM D 97. The pour point can be directly related to whether or not the oil can be poured from a container at low temperatures. Although pour point is a simple measure of wax crystal structure and low temperature viscosity, more precise and correlatable viscometric methods, such as ASTM D 3829 and ASTM D 4684, have been developed which better predict the ability of an oil to flow to the oil pump and throughout the system at low temperature. In actual practice, the oil in the crankcase will be a mixture of oil and small amounts of fuel fractions, the composition depending on several factors (see 4.3.4).

Some oils display an increase in pour point when exposed to repeated cycling at temperatures below and above the pour point. Appendix B of SAE J300 (taken from Federal Test Method, Standard No. 791C, Method 203.1) describes a procedure, commonly called the stable pour point, for evaluating the tendency of the pour point to so increase. While no longer a mandatory procedure in the low temperature classification requirements of SAE J300, the measurement of the stable pour point continues to be recommended when significant changes in formulation or base stock sources are made.

4.2.2 FLASH POINT AND FIRE POINT—The flash point of a petroleum product is the lowest temperature to which the product must be heated under specific conditions to give off sufficient vapor to form a mixture with air that can be ignited momentarily by a specified flame. Fire point is the lowest temperature to which a product must be heated under prescribed conditions to burn continuously when the mixture of vapor and air is ignited by a specified flame.

Flash and fire points are significant from the viewpoint of safety and should be related to the temperatures to which petroleum products will be subjected in storage, transportation, and use. Normally, engine oils will present no hazards in this respect but the minimum flash point that can be tolerated must be determined in each application. Flash point may also be used to indicate gross contamination of used oil by a volatile product such as gasoline or diesel fuel. Methods of obtaining this type of information are ASTM D 56, ASTM D 92, ASTM D 93, ASTM D 1310, and ASTM D 3828. ASTM D 92 and ASTM D 93 are the preferred methods for unused engine oils.

4.2.3 DISTILLATION DATA—The volatility characteristics of engine oils can be defined by distillation procedures. Because engine oils are comprised of relatively high boiling point fractions, which would thermally crack in an atmospheric distillation, a reduced-pressure (vacuum) distillation method, i.e., ASTM D 1160, must be used. ASTM D 2887, which gives boiling-range distribution data by gas chromatography, has gained acceptance and is often used in place of ASTM D 1160. ASTM D 2887 and CEC L-40-T-87 are both currently used to measure the evaporative loss or volatility of engine oils. Neither of these methods is entirely satisfactory and new methods are under development by ASTM. Correlations between performance characteristics, such as oil consumption, and the volatility characteristics of the oil in use need to be developed with actual engine tests.

4.2.4 ALKALINITY AND ACIDITY—The alkalinity or acidity characteristics of petroleum products can be measured by any one of several standardized methods. Methods currently used include ASTM D 664, ASTM D 974, ASTM D 2896, and ASTM D 4739. Changes in alkalinity or acidity with use give some indication of the nature of the changes taking place in the engine oil. For example, a reduction in base number can be ascribed to neutralization of basic additive components such as metal containing detergents as well as certain ashless dispersants. An increase in acid number may be ascribed to engine oil oxidation and/or contamination by products of combustion. Base number of a new oil is an indication of an oil's ability to resist the deleterious effects associated with high sulfur levels in diesel fuels. Different titration methods may yield different base numbers on the same oil. Therefore, caution is necessary in applying base number—oil performance relationships. For diesel engines, relationships have been published between base number of the new oil, change in base number during service, fuel sulfur content, and desired engine oil drain interval. The change in base number in service can be used under certain conditions to evaluate engine oil change interval practices. Both ASTM D 2896 and ASTM D 4739 methods are commonly used in these instances.

4.2.5 CARBON RESIDUE—The base stock components of engine oils are mixtures of many compounds that differ widely in their physical and chemical properties. Some vaporize at atmospheric pressure without leaving an appreciable residue. When destructively distilled, the nonvolatile compounds may leave a carbonaceous material known as carbon residue. Two methods used for evaluating base stocks in this respect are ASTM D 189 and ASTM D 524. ASTM D 4530 is essentially equivalent to ASTM D 189 while minimizing sample size. Engine oils containing ash-forming constituents, such as the additives commonly used in formulating oils, may give misleading high carbon residues by either method. Carbon residue has little value as a guide for predicting deposit-forming tendencies in automotive engines, but may relate to intake port deposits in certain large, two stroke cycle, natural gas fueled stationary engines.

4.2.6 ASH CONTENT—The amount of ash formed from burning engine oils may be obtained by ASTM D 482. However, ASTM D 874 is now the method most commonly used because it is a more accurate measure of ash-forming constituents. When tested by ASTM D 482, some metals are partially volatilized and lost, giving erroneously low values. The ash produced from burning new engine oils is principally related to the concentration of ash-producing additives in the oil. In addition to the additive contribution, the ash produced by used oils will also be a function of the amount of contaminants, such as lead compounds present in the engine oil if the engine is operated on a leaded gasoline. High values can also result from other contaminants, such as dirt, iron oxide, wear metals, and corrosion products. Ash forming substances in an oil may contribute to deposits on combustion chamber surfaces, spark plugs, and intake or exhaust valves, which can influence the combustion characteristics, exhaust valve sealing and certain driveability characteristics of an engine. However, the mechanism for the buildup of deposits in these areas is very complex and depends on many variables in addition to the ash content of the oil.

4.2.7 COMPATIBILITY—Engine oils are expected to be homogeneous and completely miscible with all other engine oils with which they might be mixed in service. When oils are mixed in any proportion, there should be no evidence of separation either of the additives or of the oils when the mixed oils are heated to a temperature as high as 232 °C and cooled to a temperature as low as the pour point of the mixture. The homogeneity and miscibility test currently used to evaluate engine oils is Federal Test Method Standard No. 791C, Method 3470.1.

4.2.8 FOAMING—Oils with poor antifoaming characteristics have been shown to result in decreased oxidation resistance and reduced lubricant efficacy. A bench test for determining this quality is ASTM D 892.

4.2.9 GRAVITY, COLOR, ODOR—Gravity (density) may be used to characterize the basic hydrocarbon type of the base stocks. Gravity and color are factors generally associated with the quality control of manufactured products rather than with performance characteristics. ASTM D 287 and ASTM D 1298 may be used to determine the gravity and density characteristics of oil. The color of engine oils may be specified by using ASTM D 156 or more commonly ASTM D 1500.

It is expected that engine oils will not produce offensive odors due to the nature of the base stocks or the additive agents with which the oil is compounded; nor should offensive odors or toxic vapors be generated during use of, or prolonged storage of, an engine oil. There are no standardized odor tests suitable for engine oils.

4.2.10 ELEMENTAL ANALYSIS—Elemental analysis of engine oils is often used as a means of quality control. Instrumental analytical techniques, such as emission spectroscopy, ICP, atomic absorption spectroscopy, and X-ray emission spectroscopy, are useful in this respect. Similar analyses of used oils will provide information relative to the changes in the elemental content of the engine oil. These data can also give a measure of contamination by materials such as ingested dirt, coolant, or products of combustion, especially with engines using leaded gasoline. They also can provide information relative to the extent of wear in the engine. Concentrations of the following elements are commonly determined:

a. Additive elements such as barium, boron, calcium, copper, magnesium, molybdenum, nitrogen, phosphorus, silicon, sodium, sulfur, and zinc.
b. Contaminants such as lead, silicon, chlorine, bromine, and potassium.
c. Wear metals such as aluminum, chromium, copper, iron, lead, molybdenum, and tin.

4.2.11 INFRARED ANALYSIS—Infrared spectrophotometry techniques are valuable in identifying the chemical structures found in base stocks and additives. Changes in these structures can be determined by comparing results of analyses of new and used oils. In used oils, it is also possible to measure oxidation and/or nitration, and to identify the presence of contaminants (e.g., fuel dilution and fuel soot), water, antifreeze, and similar materials.

4.2.12 FILTERABILITY—The tendency of an oil to form gels or other filter plugging material in the presence of water can be evaluated by use of the General Motors Engineering Standard GM 9099P. This method is being considered for standardization by ASTM in the near future.

4.3 Tests Pertinent to Used Oils

4.3.1 USED OIL PROPERTIES—The analysis of a used engine oil may be of value in establishing the condition of the engine and may be helpful in estimating the remaining useful life of the oil. To be of most value, used oil analyses must be taken periodically during the drain interval and a trend line established. The conditions of usage also must be considered in evaluating used oil analyses.

4.3.2 INSOLUBLE CONTENT—Insoluble materials found in both new and used engine oils may be determined using ASTM D 91, or the more frequently used ASTM D 893. Use of these methods permits an evaluation of the contaminant content and buildup of insoluble materials through oxidation, etc. However, the results must be judged with care, because minor changes in the analytical procedure can produce different results. For example, the age and purity of the coagulant solutions specified in ASTM D 893 can affect the results obtained.

With modern highly dispersant oils, the determination of insolubles has become increasingly difficult. Use of coagulant in ASTM D 893 may be required to make accurate determinations. ASTM D 4055 measures all insolubles greater in size than 0.8 mm. However, current interlaboratory precision (reproducibility) is poor.

4.3.3 COOLANT (MOISTURE) CONTENT—Small quantities of water will frequently be found in used engine oil as contamination from products of combustion, leakage from the cooling system, or condensation from atmospheric moisture. ASTM D 95 defines a process for determining the water content of used oil. For a qualitative determination, a commonly used simple test is to heat a drop of oil on aluminum foil. A snapping or crackling sound indicates free or suspended water in the oil. Cooling system leakage can be suspected when water is found in the oil on cool down after operation for several hours under high temperature conditions, such as interstate highway driving. The presence of glycol can be a more definitive indication of leakage. Glycol is detected by distillation of the aqueous material, followed by chemical analyses or infrared spectrophotometry on the distillate. A less complicated procedure which is adaptable to field kit use and gives positive, trace, or negative results is ASTM D 2982. Some additives commonly used in formulating engine oils contain glycol at a level that will give a positive result. If the new oil gives a positive result, the test in its simple form will be inadequate for detecting coolant glycol in used oils and the oil supplier should be consulted for advice.

4.3.4 FUEL DILUTION—Engines in good mechanical condition and operated at normal temperatures will usually show a small amount of fuel dilution in the used engine oil. Low operating temperatures, rich mixtures of fuel and air, and low ambient temperatures will promote fuel dilution, particularly if the engine is in poor mechanical condition or crankcase ventilation is inadequate. High dilution reduces oil viscosity and pour point. The presence of such dilution can cause accelerated wear and promote the formation of sludge, varnish, and rust. The presence of a high dilution level may indicate a need for engine maintenance. Dilution may be determined by ASTM D 322 or by ASTM D 3607. This latter method also produces a dilution-free sample for subsequent analyses. These methods are useful only with gasoline engines since the distillation range for diesel fuels in many cases overlaps that for the engine oils used in diesel engines. Procedures applicable to both diesel and gasoline engine oils are ASTM D 3524 and ASTM D 3525. For laboratories so equipped, ASTM D 2887 is also a suitable method for measuring fuel dilution.

The flash point may also be used to approximate fuel dilution. If the flash point test is utilized, it is extremely important to measure the flash point of the new (reference) oil to establish a correct baseline (see 4.2.2). The infrared spectrophotometer may also be utilized as a test tool to approximate fuel dilution in used oil. Viscosity decrease may also indicate the presence of fuel dilution; however, high levels of soot contamination can mask such reductions.

4.3.5 SOOT CONTENT—Soot content of used diesel engine oils can cause a number of problems related to additive complexing and abnormal viscosity increases. The soot is formed by the combustion process and is especially serious when combustion is incomplete. Currently soot can be detected by ASTM E 1131. A qualitative indication of soot content can be made by viscosity measurement.

5. Performance Characteristics—In the operation of an internal combustion engine, engine oils are expected to lubricate, cool, seal, maintain cleanliness, and protect against wear and corrosion. An oil's ability to perform these functions depends on the combined effectiveness of its base stock and additives, as well as operating conditions, fuel quality, and the design and the mechanical condition of the engine. Although the physical and chemical tests described in the preceding sections can be used for quality control to insure manufacturing uniformity, they are not effective for accurately defining performance characteristics at operating loads and temperatures. Only actual performance evaluations in special laboratory engines and in field tests will define the capabilities of an engine oil. Laboratory diesel and gasoline engine tests that have become industry recognized for evaluating engine oils are described in SAE J304. SAE J183 and SAE J1423 classify oils according to performance criteria based on results from engine dynamometer tests. These criteria are generally correlated with field test results. Where other operational properties are of interest, specific tests must be developed using the equipment and conditions most relevant to a given situation.

Although the laboratory engine tests are necessary and valuable aids to engine oil development and evaluation, they have limitations. In many instances, the final proof-of-performance is established by field tests of the oil in actual vehicle service. While no industry standardized field test procedures are currently available, the SAE Lubricants Review Institute has furnished some guidelines in their procedures manual. The most meaningful results on a given oil are obtained by evaluation in the most severe type of service expected to be encountered by a particular engine oil.

6. ***Handling and Disposal of Used Oils***—Continuous, long-term contact with used engine oil has caused skin cancer in laboratory animal tests. Proper protective clothing and equipment (gloves, etc.) should be used when handling used engine oil. Exposed skin areas should be washed thoroughly after exposure to used engine oil.

Clothing should be laundered regularly, especially after contact with used engine oil. In addition, proper disposal of used engine oil is very important. For more information on handling and disposal of used engine oil, contact your oil supplier, the American Petroleum Institute, or appropriate federal, state, or local government agencies.

7. ***Conclusions***—The lubrication requirements for modern engines are extremely complex. Current engine oils are the result of extensive research and development aimed at meeting these requirements. It is not the objective of this document to treat the subject in detail. Rather, the purpose of this document is to define very briefly the terms frequently encountered in discussions of engine oils and engine oil performance for those technical people not directly associated with lubricants and lubricant development. For more detailed information on these matters, the reader is referred to the technical services offered by lubricant and additive manufacturers, appropriate engine manufacturer organizations, and pertinent literature available through the Society of Automotive Engineers, Society of Tribologists and Lubrication Engineers, American Society of Mechanical Engineers, American Petroleum Institute, American Society for Testing and Materials, etc. Information directly related to this document may be found in the listed references (see Section 2).

8. *Notes*

8.1 **Marginal Indicia**—The (R) is for the convenience of the user in locating areas where technical revisions have been made to the previous issue of the report. If the symbol is next to the report title, it indcates a complete revision of the report.

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PASSENGER-CAR TYPE ENGINE OILS