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Physical and Chemical Properties of Engine Oils

RATIONALE

a. Changes to section 2.1.2 on applicable publications.

b. Changes to section 2.1.3 Other Publications.

c. Changes to section 3.1.7 on Gas-to-Liquid Synthesis.

d. Changes to section 3.2 on Additive Agents.

e. Changes to section 4.1.3 on Viscosity Index Improvers.

f. Changes to section 4.2.1 Cloud Point and Pour Point.

g. Changes to section 4.2.3 on Distillation Data.

h. Changes to section 4.2.6 on Ash Content.

i. Changes to section 4.2.7 on Compatibility.

j. Changes to section 4.2.8 on Foaming.

k. Changes to section 4.2.10 on Elemental Analysis.

l. Changes to section 4.2.12 on Filterability.

m. Changes to section 4.2.13 on Biodegradability.

n. Change the name of the Fuels and Lubricants Division to Fuels and Lubricants Council.

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.

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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 Publications

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, Tel: 877-606-7323 (inside USA and Canada) or 724-776-4970 (outside USA), www.sae.org.

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, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, Tel: 610-832-9585, www.astm.org.

ASTM DS 39b	Viscosity Index Tables for Celsius Temperatures
ASTM D 56	Test Method for Flash Point by Tag Closed Cup 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 Tester
ASTM D 93	Test Methods for Flash Point by Pensky-Martens Closed Cup 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 Products
ASTM D 156	Test Method for Saybolt Color of Petroleum Products (Saybolt Chromometer Method)
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 (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 of Petroleum Products 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	Test 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	Standard Practice for Calculating Viscosity Index from Kinematic Viscosity at 40 and 100 °C
ASTM D 2500	Test Method for Cloud Point of Petroleum Products
ASTM D 2887	Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography
ASTM D 2896	Test Method for 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 Oils by Gas Chromatography
ASTM D 3525	Test Method for Gasoline Diluent in Used Gasoline Engine Oils by Gas Chromatography
ASTM D 3607	Test Method for Removing Volatile Contaminants from Used Engine Oils by Stripping
ASTM D 3828	Test Methods for Flash Point by Small Scale Closed Cup Tester
ASTM D 3829	Test Method for Predicting the Borderline Pumping Temperature of Engine Oil
ASTM D 4055	Test Method for Pentane Insolubles by Membrane Filtration
ASTM D 4485	Standard Specification for Performance of Engine Oils
ASTM D 4530	Test Method for Determinatino of Carbon Residue (Micro Method)
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 Shear Rate and High Temperature 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 Measuring 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	Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry
ASTM D 5002	Test Method for Density and Relative Density of Crude Oil by Digital Density Analyzer
ASTM D 5133	Test Method for Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature-Scanning Technique
ASTM D 5185	Test Method for Determination of Additive Elements, Wear Metals, and Contaminants in Used Lubricating Oils and Determination of Selected Elements in Base Oil by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
ASTM D 5275	Test Method for Fuel Injector Shear Stability Test (FISST) for Polymer Containing Fluids
ASTM D 5293	Test Method for Apparent Viscosity of Engine Oils Between –5°C and –35 °C Using the Cold-Cranking Simulator
ASTM D 5800	Test Method for Evaporation Loss of Lubricating Oils by the Noack Method
ASTM D 6082	Test Method for High Temperature Foaming Characteristics of Lubricating Oils
ASTM D 6278	Test Method for Shear Stability of Polymer Containing Fluids Using a European Diesel Injector Apparatus
ASTM D 6417	Test Method for the Estimation of Engine Oil Volatility by Capillary Gas Chromatography
ASTM D 6795	Test Method for Measuring the Effect on Filterability of Engine Oils After Treatment with Water and Dry Ice and a Short (30 min) Heating Time
ASTM D 6922	Test Method for Determination of Homogeneity and Miscibility in Automotive Engine Oils
ASTM E 1131	Test Method for Compositional Analysis by Thermogravimetry
ASTM MNL 1	Manual on the Significance of Tests for Petroleum Products: 7th 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

American Society of Lubrication Engineers, 1951, “The Physical Properties of Lubricants”

API Publication 1509, latest edition, plus revisions

CEC L-14-A-93, “Evaluation of the Mechanical Shear Stability of Lubricating Oils Containing Polymers” Method

Federal Test Method Standard No. 791C, Method 203.1, “Pour Stability of Lubricating Oils”

A. J. Caines and R. F. Haycock, “Automotive Lubricants Reference Book,” Society of Automotive Engineers, 1996

A. Schilling, “Automotive Engine Lubrication,” Broseley, England: Scientific Publications (G.B.) Ltd., 1972

C. M. Georgi, “Motor Oils and Engine Lubrication,” New York: Reinhold Publishing Corporation, 1950

Dieter Klamann, “Lubricants and Related Products,” Weinheim: Verlag Chemie (F.R.G.) GmbH, 1984

G. J. Schilling and G. S. Bright, “Fuel and Lubricant Additives—II. Lubricant Additives,” Lubrication Vol 63, No. 2, 1977

George E. Totten, Steven R. Westbrook, and Rajesh J. Shah, “Fuels and Lubricants Handbook: Technology, Properties, Performance, and Testing,” American Society for Testing and Materials, ASTM Manual MNL37WCD, 2003

Jacques Denis, Jean Briant, and Jean-Claude Hipeaux, “Lubricant Properties Analysis and Testing,” Editions TECHNIP, 2000

M. Campen, D. Kendrick, and A. Markin, “Growing Use of Synlubes,” Hydrocarbon Processing, February 1982

N. Benfaremo and C. S. Liu, “Crankcase Engine Oil Additives,” Lubrication Vol 76, No. 1, 1990

R. C. Gunderson and A.W. Hart, “Synthetic Lubricants,” New York: Reinhold Publishing Corporation, 1962

William A. Gruse, “Motor Oils, Performance and Evaluation,” New York: Reinhold Publishing Corporation, 1967

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

A variety of different processes are used in the manufacture of base stocks for engine oils. These processes are briefly described in the following paragraphs:

3.1.1 Distillation

Crude oil as it comes from the ground is a highly complex mixture 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 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 distilled into a series of fractions of different viscosity and volatility. Base oils produced using the distillation process include “NEUTRALS” and “BRIGHT STOCKS” (with neutrals generally being of lower viscosity than the bright stocks).

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 850 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 1600 mm^2/s (cSt) (650 to 7400 SUS).

3.1.2 Solvent Refining

The fractions resulting from vacuum distillation contain nitrogen and sulfur 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 can be removed through separation processes using solvents such as phenol, furfural, or N-methyl pyrrolidone. This processing step is commonly referred to as solvent extraction.

Waxy materials present in the base stock 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.

3.1.3 Hydrogen Processing

Hydrogen processing can occur separately or in conjunction with solvent processing. This processing can range in severity from hydrogen treating to hydrogen cracking of base stocks. Both hydrotreated and hydrocracked base stocks are typically composed of higher percentages of saturates and reduced sulfur contents relative to solvent refined base stocks. The various modern hydrogen processes are briefly described below.

3.1.3.1 Hydrotreating

Hydrotreating converts some of the unsaturated and aromatic molecules in the base stock into saturated molecules. In addition, significant desulfurization occurs along with some nitrogen removal. Mild hydrotreating, also known as hydrofinishing or hydrofining, is normally used to improve color and odor.

3.1.3.2 Hydrocracking

The hydrocracking process changes virtually all molecules through cracking, isomerization, and saturation reactions. Aromatics are converted to naphthenes, while many of the naphthene rings are broken open. Hydrocracking can replace solvent extraction, but other processes such as deasphalting and dewaxing are still required.

3.1.3.3 Hydroisomerization

Base stocks may also be produced by isomerization of wax or high wax content fractions from the crude oil refining process. Normal paraffinic molecules are primarily being rearranged to isoparaffins, while some hydrocracking of molecules may occur. The feed for this process is normally sourced from the refinery's solvent dewaxing unit or from wax manufactured by Fischer Tropsch Process. Typically solvent dewaxing follows to meet pour point requirements.

3.1.3.4 Hydrodewaxing

Catalytic hydrodewaxing is an alternative to solvent dewaxing. Wax molecules are catalytically cracked to lower boiling-products and/or isomerized to isoparaffins. This step typically follows either a hydrocracking or solvent extraction step. Catalytic dewaxing does not produce the waxy by-products used as feed source for hydroisomerized base stocks.

3.1.4 Oligomerization

Olefin oligomers are made by linking together a low molecular weight material, such as ethylene, to produce a specific olefin. The olefins are then chemically joined ("oligomerized") to produce a base stock of suitable molecular weight and viscosity. Olefin oligomers are all hydrocarbon structures, containing no sulfur, phosphorus, or metals. When specific 1-alkenes are used as feed, these materials are commonly known as poly-alpha-olefins (PAOs).

3.1.5 Esterification

Esters are formed by chemically reacting an acid and an alcohol. The most common organic esters used as engine oil base stocks are dibasic acid esters and polyol esters. Dibasic esters, or diesters, are the reaction products of a dibasic acid, such as adipic acid, and an alcohol containing one reactive hydroxyl group, such as 2-ethylhexyl alcohol. By reacting a monobasic acid with an alcohol with two or more hydroxyl groups, polyol esters are produced. "Hindered" alcohols commonly used to produce polyol esters are trimethylolpropane (TMP) and pentaerythritol (PE).

3.1.6 Recycling and Re-Refining

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.

Re-refined base stocks may be manufactured from used oil by re-refining processes. Re-refined stocks shall be substantially free from additives and 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.

3.1.7 Gas-to-Liquid Synthesis

The technology for the conversion of gas to liquids is known as the Fischer-Tropsch (F-T) synthesis. This F-T process involves the generation of long-chain hydrocarbons from Syn-Gas (carbon monoxide and H2). Natural gas as well as coal can be used as feed products for the production of Syn-Gas. The base oils produced from this process have no detectable levels of sulfur, nitrogen, or aromatics, and they are water white. They have a very narrow hydrocarbon distribution and excellent oxidation stability characteristics.

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 15 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, 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:

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

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 improper in oil solution, the viscosity contribution of the VI improper 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 improper 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 Sequence VIII (ASTM D 6709) engine test to the new oil viscosity. Shear stability can also be measured using CEC L-14-A-93 and ASTM D 6278.

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 capillary viscometer method, ASTM D 5481, has been developed. 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. In addition to ASTM D 2500 which is a manual method, there are several automated cloud point methods; ASTM D 5771, ASTM D 5772 and ASTM D 5773.

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). In addition to ASTM D 97 which is a manual method, there are several automated pour point methods; ASTM D 5949, ASTM D 5950 and ASTM D 5985.

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. Shipping regulations of DOT and IATA require ASTM D 93 flash point data for all transportation of liquid petroleum products and lubricants.

4.2.3 Distillation Data

The volatility characteristics of engine oils can be defined by several procedures. Engine oil volatility can be related to engine oil consumption in both gasoline and diesel engines.

Distillation is one method. Because engine oils are comprised of relatively high boiling point fractions that thermally degrade at high temperatures, it is impractical to use traditional bulk distillation methods to characterize engine oils. Two different methods have been generally adopted for characterizing engine oil volatility; ASTM D 5800 and ASTM D 6417.

ASTM D 5800, *Test Method for the Evaporation Loss of Lubricating Oils by the Noack Method*, measures the amount of oil evaporated for a sample in 1 hour at 250 °C under standard conditions.

ASTM D 6417, *Test Method for the Estimation of Engine Oil Volatility by Capillary Gas Chromatography*, is very similar to the more familiar ASTM D 2887, *Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography*. From the gas chromatographic curve, the amount of oil volatility at less than 700 °F can be calculated.

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

Caution should be used when interpreting ASTM D874 data due to the inconsistency of the results. An ASTM work group is reviewing ASTM D874 with the purpose of improving the percision of the test. ASTM D974 results could be quite variable depending on the combustion conditions and the composition of the lue oil. 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 present in the engine oil. 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.

Evidence is emerging that ash content of engine oils may influence the maintenance cycle of diesel particulate filters. Work is underway to understand the relationship of engine oil ash and DPF life.

Should add caution about the inconsistency of ASTM D 874 results. They could be quite variable depending on the ignition conditions and the composition of the lube oil. Not all meallic constituents form sulfates; a number of nonstoichiometric compounds are also produced. See numerous cautions included in the ASTM D 874 test method, and details about them in: R. A. Nadkarni, R. R. Ledesma, and G. H. Via, "Sulfated Ash Test Method: Limitations of Reliability and Reporducibility," SAE paper 952548 (1995).

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 ASTM D 6922.

4.2.8 Foaming

Oils with poor antifoaming characteristics have been shown to result in decreased oxidation resistance and reduced lubricant efficacy. Bench tests for determining this quality are ASTM D 892 and ASTM D 6082. Refer to the ASTM procedures for repeatability and reproducibility data. Caution should be used when comparing foam results between labs.

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.

Frequently used methods are:

Atomic absorption spectrometry—ASTM D 4628
Inductively coupled plasma atomic emission spectrometry—ASTM D 4951 and ASTM D 5185
X-ray fluorescence spectrometry—ASTM D 4927 and ASTM D 6443