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An American National Standard

# Standard Specification for Miscibility with Gasoline and Fluidity of Two-Stroke-Cycle Gasoline Engine Lubricants[1]

This standard is issued under the fixed designation D 4682; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This specification describes four categories of lubricants intended for use in two-stroke-cycle spark-ignition gasoline engines based on their miscibility with gasoline and their low-temperature fluidity.

1.2 The following *safety hazards caveat* pertains only to the test methods described in this specification. *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.*

1.3 The values for temperature, pressure, and so forth stated in SI units are the standard.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 97 Test Method for Pour Point of Petroleum Products[2]

D 439 Specification for Automotive Gasoline[3]

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids[2]

D 874 Test Method for Sulfated Ash from Lubricating Oils and Additives[2]

D 2983 Test Method for Low-Temperature Viscosity of Lubricants Measured by Brookfield Viscometer[2]

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *fluidity—of two-stroke-cycle gasoline engine lubricants*, following industry practice, this term is used to designate the absolute viscosity in millipascal·seconds (centipoises) of the lubricant under test. In general usage, fluidity is the reciprocal of absolute viscosity.

3.1.2 *miscibility—of two-stroke-cycle gasoline engine lubricants*, an inverse function of the time required for a fuel and lubricant introduced into the apparatus as separate phases to produce a single-phase mixture by agitation under controlled conditions.

## 4. Classification

4.1 The candidate oils are classified into Categories 1 through 4 according to the temperature at which the tests are conducted; respectively, 0°C (32°F), −10°C (14°F), −25°C (−13°F), and −40°C (−40°F). Each category has its own reference oil, which is the same for both the miscibility and fluidity tests.

## 5. Qualification Requirements

5.1 *Miscibility*—When tested in accordance with Section 6, candidate oils that mix with the gasoline in not more than 110 % of the number of inversions of the apparatus required to mix the reference oil, and that do not separate on standing, qualify as miscible.

5.2 *Fluidity*—When tested in accordance with Section 7, candidate oils meet the requirements for fluidity if their viscosity is not more than 10 % higher than that of the reference oil.

## TEST METHODS

## 6. Miscibility Test Method

6.1 *Summary of Test Method*—The candidate oil and gasoline are placed as separate phases in a stoppered-glass cylinder and mixed by end-over-end rotation of the cylinder under controlled conditions at the temperature appropriate to the category of the oil.

NOTE 1—This procedure specifies that the lubricant be mixed with gasoline. Some fuels in current use are partially or predominately composed of oxygenated compounds such as alcohols, and some lubricants that mix readily with gasoline may not mix with such fuels. A variant procedure can be run to determine the ability of a lubricant to mix satisfactorily with a fuel consisting partially or wholly of oxygenates. In this case, the miscibility test must be run using the candidate oil in the oxygenate or oxygenate-containing fuel against the reference oil in gasoline.

6.2 *Significance and Use*:

6.2.1 The lubricants used in two-stroke-cycle gasoline engines normally reach the surfaces to be lubricated as a mixture

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] Discontinued; see *1990 Annual Book of ASTM Standards*, Vol 05.01.

with the fuel. This mixture can either be prepared in advance, usually in the engine fuel tank, or can be produced by the metered injection of oil into the fuel stream at some point before its entry into the engine crankcase. In either case, the fuel and lubricant shall be readily miscible, and if the lubricant is metered into the fuel stream, it must be readily pumpable. As it cannot be known in advance whether a given lubricant will be mixed with the fuel in advance or be injected into the fuel stream by a metering pump, both miscibility and fluidity shall be determined.

6.2.2 The temperature at which the miscibility and fluidity of an oil is determined does not necessarily reflect the expected temperature of use. For example, outboard motor manufacturers normally require the use of oils of Category 3, which are tested at −25°C (−13°F), even though outboards are rarely used at such temperatures. The reason is that Category 3 oils are readily pumpable and will mix quite rapidly with gasoline at temperatures above about 5°C (40°F) with little or no agitation. This is an important consideration for boats with outboard motors using a fuel-oil mix that is made up in large built-in tanks which cannot readily be stirred or shaken.

6.3 *Apparatus*:

6.3.1 *Rotator*—This consists of three or four standard apparatus clamps to carry the cylinders specified in 6.3.2 mounted on a horizontal shaft of about 12- to 14-mm (0.4- to 0.6-in.) diameter and about 300 mm (12 in). long mounted between antifriction bearings, driven by an electric motor, and provided with a revolution counter. The shaft is rotated at about 10 to 14 r/min so that the cylinders are continuously being inverted as the shaft rotates. A maximum of three candidate oils can be run against one reference oil. While it would be possible to design apparatus to handle a greater number of samples, four appears to be a practical limit for ease of operation. In Fig. 1 a photograph of a suitable rotator is shown.

6.3.2 *Four (or Fewer) Graduated Cylinders, 500 mL*, capable of remaining securely stoppered in any attitude. The length-to-diameter ratio of the cylinders may be in the range from 10 to 12:1, but all cylinders used in the same test shall be identical within normal commercial glassware tolerance.

6.3.3 *Stoppered Flask* of about 500-mL capacity for each graduated cylinder.

6.3.4 *Freezer* capable of maintaining a temperature controllable within ±1°C (2°F) in the range from 0 to −40°C (32 to −40°F). The freezer shall be provided with a transparent cover and be capable of accommodating the complete rotator assembly while in operation, together with space for the storage of additional cylinders and flasks. A cover with two panes of glass or plastic separated by an air space of about 10 to 15 mm (0.4 to 0.6 in.) is advised.

6.4 *Reagents and Materials*:

6.4.1 *Reference Oils*—A different reference oil is required for each miscibility/fluidity category. These are: for Category 1, ASTM reference oil VI-GG;[4] for Category 2, ASTM reference oil VI-FF;[5] for Category 3, ASTM reference oil VI-D;[6] and for Category 4, ASTM reference oil VI-II.[5] See Annex A1 for information on the composition and properties of these oils.

6.4.2 Any full-boiling-range gasoline meeting the general requirements of Specification D 439 volatility Classes A, B, or C can be used. When this procedure is run in conjunction with an engine performance test, it is the normal practice to use the gasoline specified for the performance test. Oxygenate blends and other fuels containing nonhydrocarbon blending components shall not be used.

6.5 *Calibration and Standardization*—As a reference oil is tested simultaneously with each set of candidate oils, no other standardization procedure is required.

6.6 *Procedure*:

6.6.1 Approximately 25 mL of the oil to be tested and 450 mL of gasoline shall be available for each sample to be run, including the reference oil.

6.6.2 Pour 19 to 21 mL each of the reference oil and of the candidate oil(s) into separate 500-mL mixing cylinders and insert their stoppers.

6.6.3 It is preferred, but not mandatory, to purge the cylinders with nitrogen before inserting the stoppers.

6.6.4 Prepare one 395- to 405-mL sample of gasoline in a stoppered flask for each oil sample to be tested, including the reference oil.

6.6.5 Place the cylinders and the gasoline samples with the rotator into a freezer at the required test temperature for a minimum of 16 h. The cylinders may be attached to the rotator or may be stored separately in the freezer at this time.

6.6.6 At the end of the soak period (if this has not already been done), mount the mixing cylinders onto the rotator at about 30° to the vertical, clamping them at about the 350-mL mark.

6.6.7 Remove the stopper and empty one of the gasoline samples into each cylinder in turn, pouring carefully down the side so as to minimize mixing. Replace and secure the stopper of each cylinder as soon as it has been filled.

6.6.8 When all cylinders are charged with gasoline, replace the freezer cover and start the rotator.

6.6.9 Watch the cylinders, and record for each the number of revolutions required for complete mixing of the gasoline and oil (no unmixed oil visible on the bottom with the cylinder upside down). If difficulty is experienced with fogging of the transparent freezer cover, proprietary antifogging compounds are usually effective.

6.6.10 After the test is completed, leave the cylinders in the freezer in the upright position for a 48 h minimum and check for phase separation. The cylinders may be left in place or may be removed from the rotator during this portion of the test.

6.6.11 Any candidate oil that requires over 10 % more revolutions to mix than is required by the reference oil or that

[4] The sole source of supply of the reference oil known to the committee at this time is Lubrizol Corp., 29400 Lakeland Blvd, Wickliffe, OH 44092. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.

[5] *Annual Book of ASTM Standards*, Vol 05.02.

[6] The sole source of supply of the reference oil known to the committee at this time is Citgo No. 93734 from Citgo Petroleum Corp., 555 E. Butterfield Rd., Lombard, IL 60148. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.

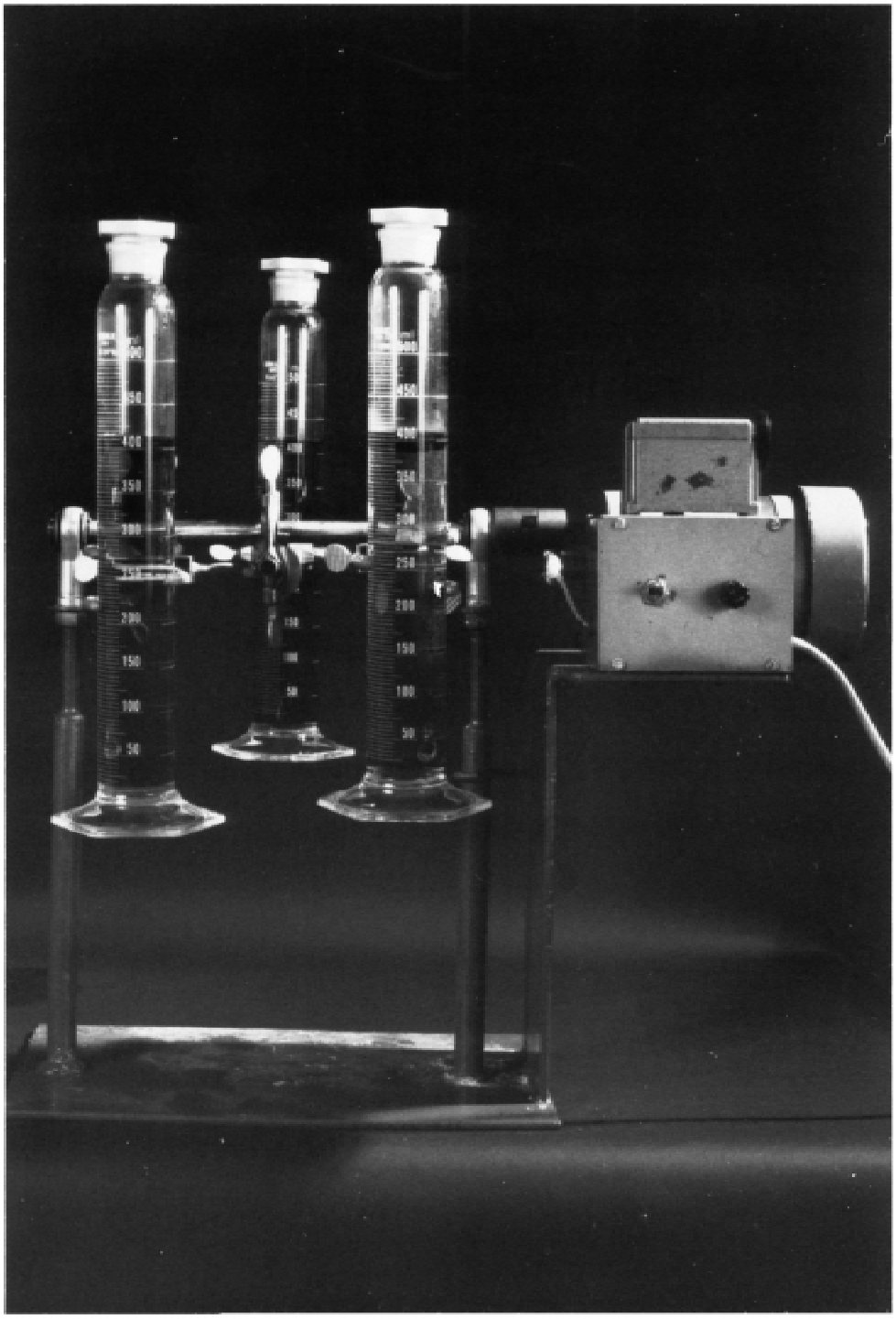

**FIG. 1 Rotator**

separates from the gasoline on standing fails. Otherwise pass.

6.7 *Report*—Report the temperature at which the test was conducted and the number of inversions (complete rotations of the rotator shaft) required to produce mixing of the candidate oil and of the reference oil used, which must be identified. Note the category assigned. If separation is observed, report the degree of separation and the number of hours standing.

6.8 *Precision and Bias*—No statement is made about either the precision or the bias of the miscibility test, as no accepted method is available to determine these in the case of pass-fail results.

## 7. Fluidity Test Method

7.1 *Summary of Test Method*—The viscosity of the candidate oil is determined by Test Method D 2983 (Brookfield viscometer) at the same temperature as used for the miscibility test, and compared with that of the reference oil. See 3.1.1 regarding the use of the word *fluidity* in this context.

NOTE 2—The fluidity test is quite frequently used as a screen or as a control check for miscibility. The miscibility-fluidity relationship must be determined experimentally for any specific lubricant, but in most cases it will be found that a candidate oil will not qualify in a miscibility category if it does not meet the fluidity requirements of that category. For mixtures involving oxygenate-containing fuels or nonhydrocarbon lubricants such as esters or polyglycols, the low-temperature viscosity cannot be used safely to predict miscibility, but can be used as a control after the ability of the components to mix satisfactorily has been established.

7.2 *Significance and Use*—See 6.2.

7.3 *Apparatus*—The Brookfield viscometer and its associated equipment are described in Section 6 of Test Method D 2983.

7.4 *Reagents and Materials*—See 6.4.

7.5 *Calibration and Standardization*—As the results obtained from the Brookfield viscometer are critically dependent on its correct setup and functioning, the reference oil appropriate to the temperature of the test shall be run immediately before or after, preferably both before and after, any candidate oils tested. If the viscometer is being used on a continuing basis for control purposes, a reference oil check shall be made at frequent and regular intervals.

7.6 *Procedure*:

7.6.1 About 50 mL of oil is required for each test.

7.6.2 Use the procedure of Test Method D 2983 to determine the viscosity of the oil under test at the temperature appropriate to the category desired, or at the lowest standard test temperature above the pour point of the oil if the category of the candidate is unknown. The revolutions per minute of the Brookfield viscometer shall be reported, and shall be set in accordance with Section 8 of Test Method D 2983.

7.6.3 The viscosity of the candidate oil shall be compared with that of the reference oil for the category run at the appropriate temperature immediately before or after the candidate oil test under essentially identical conditions, preferably in the same viscometer. If the reference oil viscosity is determined both before and after that of the candidate oil and the values are within the repeatability range of Test Method D 2983 (Section 13), either may be used. The reference oil viscosities given in Annex A1 are approximate and for information only.

7.7 *Report*—Report the Brookfield viscosity of the candidate oil and of the reference oil, the temperature(s) at which they were determined, one of which must be the same as that at which the miscibility test was run, and the revolutions per minute used for each determination.

7.8 *Precision and Bias*—The precision and bias of this test method are as specified for Test Method D 2983.

## ANNEX

### (Mandatory Information)

### A1. REFERENCE OILS

A1.1 Information on the composition and typical physical properties of the reference oils used in this procedure is listed in Table A1.1. Note that 1 centistokes (cSt) = $10^{-6}\cdot m^2/s$, the unit of kinematic viscosity, and 1 centipoise (cP) = 1 millipascal second (mP·s), the unit of absolute viscosity. The Test Method D 2983 (Brookfield) viscosities given are approximate maximum values for information only, and must not be used as pass-fail criteria in the fluidity test.

**TABLE A1.1 Physical Properties of the Reference Oils**

| Properties | VI-GG | VI-FF | VI-D | VI-II |
|---|---|---|---|---|
| Reference oil for category | 1 | 2 | 3 | 4 |
| Viscosity, cSt at 100°C D 445 | 13.8 | 9.4 | 6.3 | 4.9 |
| Viscosity, D 2983 (Brookfield) | | | | |
| cP at −40°C | — | — | solid | 13 500 |
| cP at −35°C | — | — | — | 5300 |
| cP at −25°C | — | — | 7500 | — |
| cP at − 10°C | — | 2600 | 950 | — |
| cP at 0°C | 3250 | — | 500 | — |
| Pour point °C D 97 | −18 | −18 | −30 | −51 |
| Sulfated ash, mass % D 874 | 0.17 | 0.17 | <0.005 | 0.17 |
| Composition, vol % | | | | |
| 550 Neutral | — | — | — | 43.4 |
| 650 Neutral | 86.8 | 78.9 | 61.45 | — |
| 1400 | — | — | — | 17 |
| 150 Bright Stock | 8.0 | 8.0 | 9.00 | 9 |
| Stoddard solvent | 0.0 | 8.0 | 20.00 | 25 |
| Pour depressant | 0.2 | 0.1 | 0.2 | 0.4 |
| Other additives | 5.0[A] | 5.0[A] | 9.35[B] | 5.2[A] |

[A] Mixture of organo-metallic detergent and ashless dispersant.
[B] Primarily an ashless dispersant.