Designation: D 5770 – 02

An American National Standard

Standard Test Method for Semiquantitative Micro Determination of Acid Number of Lubricating Oils During Oxidation Testing[1]

This standard is issued under the fixed designation D 5770; 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 test method is a semiquantitative micro method intended for monitoring the changes in acidic constituents occurring in lubricating oils during oxidation testing, when the acid number of such oils falls within the range from 0.02 to 1.0 mg of potassium hydroxide per gram of sample. It is applicable to such oils as turbine oils, hydraulic oils, and other circulating oils.

NOTE 1—This test method is a micro version of Test Method D 974 and it produces results similar to that method.

1.2 This test method is designed for use where sample size is limited. It shall not be used as a replacement for higher precision methods such as Test Methods D 974 or D 664. It shall not be used to monitor oils in-service.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration[2]

D 943 Test Method for Oxidation Characteristics of Inhibited Mineral Oils[2]

D 974 Test Method for Acid and Base Number by Color-Indicator Titration[2]

D 3339 Test Method for Acid Number of Petroleum Products by Semi-Micro Color Indicator Titration[3]

D 4871 Guide for Universal Oxidation/Thermal Stability Test Apparatus[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *acid number, n*—the quantity of base, expressed in milligrams of potassium hydroxide per gram of sample, that is required to titrate a sample dissolved in a specified solvent to a specified endpoint.

3.1.1.1 *Discussion*—In this test method, the acid number is calculated from the number of drops required to produce a change in solution color from blue-green to orange, compared to the number of drops required to produce an identical color change using a reference standard. Because this is a direct comparison method, the acid number value can be reported in milligrams of potassium hydroxide per gram of sample.

4. Summary of Test Method

4.1 A 2.0-mL portion of the titration solution is titrated with a sample using a dropping pipet. The number of drops of sample required to turn the blue-green titration solution to a persistent orange color is noted.

4.2 A second 2.0-mL portion of the titration solution is titrated with an acid number reference solution of known acid number. The number of drops of the reference solution required to turn the blue-green titration solution to a persistent orange color is noted.

4.3 The estimated acid number of the sample is calculated using the acid number of the reference solution and the ratio of the number of drops of the reference solution required to effect the color change to the number of drops of the sample required for the same change.

5. Significance and Use

5.1 This test method provides a semiquantitative estimate of the acid number of lubricating oils obtained from laboratory oxidation tests using smaller amounts of sample than Test Methods D 974, D 664, or D 3339. It has specific application in Test Method D 943 and in Test Method D 4871. This test method, therefore, provides a means of monitoring the relative

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.06.0A on Chemical Analysis.

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

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

***A Summary of Changes section appears at the end of this standard.**

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oxidation of lubricating oils by measuring changes in acid number, at different time intervals and under various oxidizing test conditions.

5.2 Since this test method is semiquantitative, each laboratory shall develop its own criteria for each oxidation test method for determining when to switch from this semiquantitative test method to a more precise test method for acid number.

6. Apparatus

6.1 *Glass Beakers*, 10-mL capacity, or glass vials, 4 or 6 dram.

6.2 *Glass Syringe*, 2-5 mL capacity. A 5-mL interchangeable syringe with a 20 gage, 12 cm needle is satisfactory.

6.3 *Dropping Pipet*, glass with rubber bulb, capable of delivering 35 to 40 drops of oil.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless indicated otherwise, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Other grades can be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Acid Number Reference Solution*—Any stable, oil-soluble acid which will produce an acid number of approximately 0.5 mg KOH/g is acceptable. This solution shall be standardized using Test Method D 974 or D 664. A solution of 0.20 % mass dodecenylsuccinic anhydride in HVI 250 base oil or in an ISO 10-22 (60-100 SUS) oil has proven satisfactory.

7.3 *Alcoholic Potassium Hydroxide Standard Solution (0.1 M)*—Prepare approximately 0.1 M solution by dissolving KOH in propanol-2-ol. Standardize against pure potassium acid phthalate in about 100 mL of water, using phenolphthalein to detect the end point. (Commercial grades of this reagent have been found to be satisfactory.) (**Warning**—Corrosive.) (**Warning**—Poisonous if ingested, alkaline, causes irritation producing dermatitis.)

7.4 *Dodecylsuccinic Anhydride*—(**Warning**—Irritating to eyes and skin.)

7.5 *p-Naphtholbenzein Indicator Solution*—The p-naphtholbenzein must meet the specifications in Appendix X1 of Test Method D 974. Prepare a solution containing 10 g of p-naphtholbenzein/L in a 50:50 mixture of toluene:propanol-2-ol. (**Warning**—Flammable. Vapor harmful.)

NOTE 2—In a 1992 study, only Kodak, Fisher, and Baker (Mallinkrot) were found to meet the specifications. However, the Kodak brand is no longer available. The Fisher Reagent Solution was the only commercially available solution to meet the specifications.

7.6 *Propanol-2-ol (Isopropyl Alcohol)*—(**Warning**—Flammable.) (**Warning**—It has been reported that, unless inhibited against it, peroxides can form in propanol-2-ol and, as the storage vessel or equipment such as a dispensing bottle become empty and approach dryness, an explosive mixture can occur.)

7.7 *Titration Solution*—Mix 350 mL of toluene, 350 mL of propanol-2-ol, and 7 mL of the p-naphtholbenzein solution in a 1-L plastic bottle. Add 15 mL of 0.1 *M* potassium hydroxide alcoholic solution and mix well. (**Warning**—Flammable. Vapor harmful. Corrosive.)

NOTE 3—The strength of the titration solution depletes with time and should be periodically replaced. Up to one month has been found to be satisfactory.

7.8 *Toluene*—(**Warning**—Flammable. Vapor harmful.)

8. Procedure

8.1 *Sampling*—Withdraw the minimal amount of the test oil from the oxidation test cell according to the procedure specified in the oxidation test method.

NOTE 4—As oxidized oils can change appreciably in storage, samples should be tested as soon as possible after removal from the oxidation test equipment.

8.2 Using the syringe, measure 2.0 mL of the titration solution into a 10-mL beaker or vial.

8.3 Using the dropping pipet, add the oil sample to the titration solution one drop at a time, swirling constantly.

8.4 Count the number of drops of oil sample required to change the titration solution color from blue-green to orange. Consider the titration complete when the orange color is stable for a minimum of 10 s.

8.5 Repeat 8.2 through 8.4 using the acid number reference solution in place of the oil sample as the titrant of 2.0 mL of titration solution, so that the final colors for the two beakers match.

9. Calculation and Report

9.1 Calculate the acid number of the oil sample as follows:

$$\text{acid number, mg KOH/g} = \frac{A \times B}{C} \quad (1)$$

where:

A = number of drops of the acid number reference solution in 8.5,
B = acid number of the acid number reference solution, and
C = number of drops of the oil sample (8.4).

9.2 Report the result as follows:

$$\text{acid number, (Test Method D 5770)} = \text{(result)} \quad (2)$$

10. Precision and Bias

10.1 *Precision*—The precision of this test method was determined by statistical examination of interlaboratory test results over the acid number range from 0.06 to 1.1 mg KOH/g for nine samples analyzed by eight laboratories.[5]

[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1356.

10.1.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$0.20 \times \text{acid number} \quad (3)$$

10.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$0.70 \times \text{acid number} \quad (4)$$

10.2 *Bias*—The procedures in this test method have no bias because the acid number can be defined only in terms of the test method.

11. Keywords

11.1 acid number; color indicator titration; oxidation testing; semiquantitative

SUMMARY OF CHANGES

Subcommittee D02.06.0A has identified the location of selected changes to this standard since the last issue (D 5770–96) that may impact the use of this standard.

(1) Defined acid number more explicitly.
(2) Added warning statement to 7.6 concerning possible peroxides in propanol-2-ol.
(3) Updated Note 2 concerning *p*-naphtholbenzein indicator.

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