Designation: D 2070 – 91 (Reapproved 2001)

An American National Standard

Standard Test Method for Thermal Stability of Hydraulic Oils[1]

This standard is issued under the fixed designation D 2070; 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[2] is designed primarily to evaluate the thermal stability of hydrocarbon based hydraulic oils although oxidation may occur during the test.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *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 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

2.2 *Copper Development Association Standard*

UNS C11000 Electrolytic Tough Pitch Copper[4]

2.3 *American Iron and Steel Institute Standard (AISI)*

W-1 Carbon Tool Steel[5]

3. Summary of Test Method

3.1 A beaker containing test oil, copper and iron rods is placed in an aluminum block in an electric gravity convection oven for 168 h at a test temperature of 135°C. At the completion of the test, the copper and steel rods are rated visually for discoloration and the oil is analyzed for the quantity of sludge.

4. Significance and Use

4.1 Thermal stability characterizes physical and chemical property changes which may adversely affect an oil's lubricating performance. This test method evaluates the thermal stability of a hydraulic oil in the presence of copper and steel at 135°C. Rod colors are the evaluation criteria. Sludge values are reported for informational purposes. No correlation of the test to field service has been made.

5. Apparatus

5.1 An aluminum block with equally spaced holes is used. An example is described in Fig. A1.1 of Annex A1.

5.2 Electric gravity convection oven capable of maintaining the aluminum block at a test temperature of 135°C ± 1°C.

5.2.1 Calibrated thermocouple and temperature indicator centered in aluminum block.

5.3 250 mL Griffin beakers of borosilicate glass.

5.4 Copper test specimens are to be UNS C11000, 99.9 % pure electrolytic tough pitch copper, 6.35 mm in diameter by 7.6 cm in length (0.25 in. by 3.0 in.).

5.5 Steel test specimens are to be AISI W-1 1 % carbon steel, 6.35 mm in diameter 7.6 cm in length (0.25 by 3.0 in.).

5.6 Silicon carbide abrasive 320 grit with cloth backing.

5.7 Crocus cloth.

5.8 No. 41 Whatman filter paper,[6] 47 mm diameter.

5.9 Millipore filter,[7] 8 micron Type SC, 47 mm diameter.

5.10 Millipore glass filter holder, 47 mm, Cat #XX10.04700 or equivalent.

5.11 Cincinnati Milacron color chart.[8]

5.12 25 mL pipette.

6. Reagents

6.1 *Reagent Grade Heptane*—(**Warning—** Flammable. Health hazard.)

6.2 *Reagent Grade Acetone*—(**Warning—** Flammable. Health hazard.)

7. Preparation of Apparatus

7.1 Handle the rods at all times using forceps or clean cotton gloves.

7.2 Catalyst preparation—Clean the iron and copper catalyst rods, whether new or previously used, prior to use. Clean the rods with the 320 silicon carbide abrasive cloth while rotating the rods in a drill chuck at 1700 to 1800 r/min. Clean the surface until it has a bright copper or steel appearance. Discard rods when diameter is less than 6.2 mm.

[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.N0.08 on Stability.

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[2] This procedure was adopted from the Cincinnati Milacron Thermal Stability Test, Cincinnati Milacron Manual 10-SP-89050.

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

[4] Available from Copper Development Assoc., 2 Greenwich Office Park, Box 1840, Greenwich, CT 06836.

[5] Available from AISI, 1133 15th St., NW, Washington, DC 20005.

[6] Whatman Int. Ltd., Maidstone, England.

[7] Millipore Filter Corp., Bedford, MA.

[8] Cincinnati Milacron, 4701 Marburg Ave., Dept 97B Lubricants and Tribology, Cincinnati, Ohio, 45209, 513-841-8660.

7.3 Prepare surface finally with a crocus cloth. Remove all grind marks. Finish the rods to a lightly polished surface finish.

7.4 Wash the rods individually with acetone and air dry on completion of the polishing operation.

8. Procedure

8.1 Place a representative 200-mL sample of test oil obtained per D4057 – 88 sampling procedure in a clean 250-mL Griffin beaker containing one each of the cleaned and polished iron and copper rods.

8.2 Place the rods totally below the surface of the oil and crossed. Place them in contact with each other at one point only.

8.3 Place the beaker and its contents in the pre-heated aluminum block test fixture in the oven.

8.4 Maintain the test fixture at 135°C ± 1°C for 168 h. Start the time when the test sample is placed in the oven.

8.5 Keep the oven doors closed during the entire test period. Monitor the temperature continuously via thermocouple in the center of the test block.

8.6 At the completion of 168 h, remove the beakers from the oven and allow to cool to room temperature before proceeding. Individually remove the rods from the oil sample. Remove any loose sludge clinging to the rods with a plastic or rubber policeman and return the sludge to the oil.

8.7 *Copper Rod Analysis*—Wash the rod with heptane to remove all oil and allow to air dry. Discard the heptane wash. Make a visual evaluation of the condition of the rod against the Cincinnati Milacron color chart (available from Cincinnati Milacron[7]) and record.

8.8 *Steel Rod Analysis*—Wash the steel rod with heptane to remove all oil and allow to air dry. Discard the heptane wash. Make a visual evaluation of the rod against the Cincinnati Milacron color chart and record.

8.9 For each sample, dry a #41 Whatman filter for 1 h in an oven at 70°C and cool in a dessicator. Weigh to the nearest 0.1 mg. Vacuum filter at a nominal 200 mm of Hg vacuum oil through the pre-weighed #41 Whatman filter. Do not rinse the beaker at this time. Remove the oil filtrate and set aside. Replace the filter flask with a clean one and wash all remaining residue from the beaker with heptane. Wash the residue on the filter paper with heptane until all evidence of oil is removed. Oven dry the residue and filter paper at 70°C, 1 h, allow to cool and weigh to nearest 0.1 mg. For each sample pre-weigh an 8 micron Millipore filter pad to the nearest 0.1 mg. From the oil filtrate, pipet 25 mL of oil and vacuum filter at a nominal 200 mm of Hg vacuum through the pre-weighed 8 micron Millipore filter pad. Wash residue with heptane, air dry, and weigh to the nearest 0.1 mg.

9. Calculation

9.1 *Total Sludge Determination*—The weight of the sludge on the No. 41 Whatman paper is reported as milligrams per 100 mL of oil. Therefore, the weight of the original filter paper is subtracted from the dried filter paper plus residue and the difference divided by two. The weight of the sludge on the 8 micron Millipore filter pad is also reported as milligrams per 100 mL. The weight of the original filter pad is subtracted from the weight of the dried residue plus filter pad and the difference multiplied by four. Total sludge is the summation of the sludge from the No. 41 Whatman filter paper plus the sludge from the 8 micron filter pad. Weight of total sludge (mg/100 mL of oil)

$$T - W \times 0.5 + M \times 4 \quad (1)$$

where:

W = weight of sludge on Whatman filter in mg
M = weight of sludge on 8 micrometer millipore filter in mg
T = total weight of sludge in mg/100 mL

10. Report

10.1 Report the color of the copper and steel rods as previously determined.

10.2 Report the total sludge in mg/100 mL oil.

11. Precision and Bias

11.1 The precision of this test method was determined by a statistical analysis of interlaboratory test results. The following criteria should be used for judging the acceptability of data.

11.2 *Repeatability*—The difference between successive 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:

Copper rod color	– 1 unit
Steel rod color	– 1 unit
Total sludge	– 1.04 (X + 1)

where X denotes mean value.

11.3 *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, exceed the following values only in one case in twenty:

Copper rod color	– 4 units
Steel rod color	– 2 units
Total sludge	– 3.25 (X + 1)

where X denotes mean value.

11.4 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure, bias has not been determined.

12. Keywords

12.1 thermal stability; hydraulic oils; oil sludging; copper corrosion; steel corrosion; Cincinnati Milacron; heat test