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An American National Standard

Standard Guide for Universal Oxidation/Thermal Stability Test Apparatus[1]

This standard is issued under the fixed designation D 4871; 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 guide describes an apparatus used to measure the oxidation or thermal stability of liquids by subjecting them to temperatures in the range from 50 to 375°C in the presence of air, oxygen, nitrogen, or other gases at flow rates of 1.5 to 13 L/h, or in the absence of gas flow. Stability may be measured in the presence or absence of water or soluble or insoluble catalysts. Gases evolved may be allowed to escape, condensed and collected, or condensed and returned to the test cell.

1.2 *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 91 Test Method for Precipitation Number of Lubricating Oils[2]

D 156 Test Method for Saybolt Color of Petroleum Products (Saybolt Chromometer Method)[2]

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)[2]

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

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

D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)[2]

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

D 5770 Test Method for Semi-quantitative Micro Determination of Acid Number of Lubricating Oils During Oxidation Testing[4]

3. Summary of Guide

3.1 An apparatus is described in which a sample of test fluid, typically from 100 ml or 100 g, is subjected to thermal or oxidative degradation or both. Insoluble or soluble catalyst may be added. Gas may be bubbled through the liquid to provide agitation or to promote oxidation or both. Water or water vapor may be added. At the end of the test or at intervals throughout the test, the liquid is monitored for change in neutralization number, viscosity, weight loss, formation of sludge, or for other parameters. The corrosivity of the fluid toward any catalyst metals can be determined from the appearance and weight change of the metal test specimens, if present, or by monitoring the oil and any sludge or water for metal content. The test is terminated after a fixed time period or when a selected parameter reaches a condemning value.

NOTE 1—The volume of liquid at test temperature should be sufficient to cover the catalysts and should not extend beyond the heated portion of the bath.

4. Significance and Use

4.1 This standard describes an apparatus that provides the versatility required to conduct oxidation or thermal stability tests on liquids using a wide variety of test conditions. It is sufficiently flexible so that new test conditions can be chosen in response to the changing demands of the marketplace.

5. Apparatus [5]

5.1 *Heating Block*, as shown at the lower right in Fig. 1, to provide a controlled constant temperature for conducting tests.

5.1.1 Test cells are maintained at constant elevated temperature by means of a heated aluminum block which surrounds each test cell.

5.1.2 Holes in the aluminum block to accommodate the test cells shall provide 1.0 mm max clearance for 38-mm outside diameter glass tubes. The glass test cells shall fit into the block to a depth of 225 ± 5 mm.

NOTE 2—The original test blocks were made with spaces for ten test cells. Blocks with different number of holes are acceptable if other requirements are met.

5.1.3 The heating system shall be geometrically and thermally balanced. For thermal balance, sizes and locations of the heaters are proportioned against heat losses.

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

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

[4] *Annual Book of ASTM Standards*, Vol 05.03.

[5] A standard commercial apparatus has been found satisfactory for the purpose of this guide. This apparatus, including heating block, temperature control system, flow control system and glassware, is available from Falex Corp., 1020 Airpark Drive, Sugar Grove, IL 60554. Glassware for the Universal Oxidation test apparatus is also available from W. A. Sales, Ltd., 419 Harvester Court, Wheeling, IL 60090.

FIG. 1 Universal Oxidation Test Apparatus

5.1.4 The block is cylindrical and constructed from forged aluminum. The block has a minimum thickness of 38 mm of insulation on all sides, top and bottom. An insulation of thermally efficient ceramic fiber material is suggested.

5.1.5 The exterior jacket, sides and top are stainless steel or equivalent.

5.1.6 The block is equipped with a well for a thermocouple for temperature control and measurement, and a thermometer well for temperature calibration.

5.2 *Temperature Control System*, as shown at lower left in Fig. 1, to maintain the heating block at a set temperature.

5.2.1 The temperature controller shall be capable of maintaining the block temperature within ±0.5°C of the desired test temperature for the duration of the test. The preferred controller shall have proportional and integral control modes, and a heater malfunction alarm.

5.2.2 The range for operation is from at least 50°C to 375°C. (**Warning—**An adjustable deviation alarm that automatically shuts down the system if temperature varies outside preset limits is desirable as a safety feature and to avoid erroneous test results. A separate adjustable high temperature monitor and shutoff is desirable as a safety device.)

5.2.3 Temperature control and uniformity is the most important parameter affecting test result precision. Therefore, the heating system design is critical. Temperature from hole-to-hole and at all sides of each hole in the block shall be uniform within the 0.5°C tolerance of the total system.

5.3 *Gas Flow Control System*, as shown in Fig. 1, to provide air or other gases to each test cell.

5.3.1 A gas flow controller is required for each test cell, to provide air or other desired gases. (**Warning—**If reactive gases are to be used in the test procedure, all fittings in the gas control system must be compatible with these gases.)

5.3.2 The standard gas flow range shall be from 1.5 to 13 L/h. Flowmeters shall have a scale length sufficiently long to permit accurate reading and control to within ±5 % of full scale. Floats and tubes may be interchangeable for alternate gas flow ranges.

5.3.3 The system shall have a pressure regulator to provide constant inlet pressure and 10 flow indicators with individual, integral needle valves with regulating stems for flow adjustment.

5.3.4 The total system accuracy shall meet or exceed the following tolerances:

Inlet pressure regulator within 0.34 kPa (0.05 psig) of setpoint;

Total flow control system reproducibility within 7 % of full scale;

Repeatability of measurement within 0.5 % of full scale.

5.4 *Alternative Designs*—The equipment in 5.1-5.3 represents a preferred configuration. Alternative apparatus designs for sample heating, and for temperature and flow control shall be acceptable providing they are shown to maintain temperature and gas flow within the specified limits.

5.5 *Glassware* (Note 3), is used to contain the test sample, deliver gas into the sample, and collect, carry off or return condensable volatiles.

NOTE 3—Not all glassware is used in a single test procedure.

5.5.1 *Test Cell*, borosilicate glass, standard wall; 38-mm outside diameter, 300 ± 5-mm length, with open end fitted with a 34/45 standard-taper, ground-glass outer joint (Note 4). See Fig. 2.

NOTE 4—Initial lots of glassware were made with 45/50 standard-taper, ground-glass joints. These are also acceptable for test work; however, the 34/45 joints are preferred.

5.5.2 *Condenser, Allihn-Type*, borosilicate glass, 330± 5-mm jacket, 34/45 standard-taper ground-glass inner joint on lower end. Upper opening must admit gas delivery tube. See Fig. 2.

5.5.3 *Gas Inlet Tube I*, 8-mm outside diameter, 850 ± 5 mm long, lower end with fused capillary 1.5 to 3.5 mm inside diameter. The capillary bore shall be at 15 ± 1 mm long. The lower tip is cut at a 45° angle. The gas inlet tube may optionally include a glass ring 15 mm in diameter to support washer-type catalysts, or the support ring (see 5.5.8), from which catalyst specimens may be suspended. The position of the ring on the gas inlet tube shall be specified in the test method requiring such catalyst support. The gas inlet tube I is illustrated in Fig. 2 with a glass ring suitable for support of washer-type catalyst.

5.5.3.1 The 850-mm tube is required for testing configurations using the condenser and sampling head or the intermediate head in order that the tube extend beyond the top of the condensor so that a gas supply hose can be attached. A tube of shorter length may be substituted when other assemblies are used, provided it is long enough to attach the gas supply.

5.5.4 *Gas Inlet Tube II*, 8-mm outside diameter 455 ± 5 mm long, lower tip cut at 45° angle. The top 50 mm of the tube may optionally be bent at a 90° angle. See Fig. 2.

5.5.5 *Basic Head*, with 34/45 standard-taper, ground-glass inner joint, opening for gas inlet tube, septum port for sample withdrawal, and exit tube to conduct off-gases and entrained vapors. Overall length shall be 125 ± 5 mm. The head shall have an adapter by which an air-tight seal may be made with the gas inlet tube.[6] The septum port is preferably designed to accept a flat polytetrafluoroethylene (PTFE)-faced silicone septum such as used with gas chromatographic instrumentation. See Fig. 3.

5.5.6 *Intermediate Head*, with standard-taper inner joint at bottom for test cell and outer joint at top for condenser. Overall length shall be 170 ± 5 mm. The outlet for removal of off-gases and condensed liquids is located far enough below the gas-escape holes to ensure that condensed liquid will exit through the side tube rather than returning to the test cell. A septum port (same as in 5.5.5) is provided for sample withdrawal. See Fig. 3.

5.5.7 *Sampling Head*, with standard-taper inner joint at bottom for test cell and outer joint at top for condenser. Overall length shall not exceed 175 mm. A septum port (same as in 5.5.5) is provided for sample withdrawal. See Fig. 3.

5.5.8 *Support Ring*, 9.5-mm inside diameter, 12.7-mm outside diameter, 7 mm long, to fit on inlet tube I fitted with

[6] A Wheaton-type thermometer adapter is suitable, with an O-ring seal. All wetted parts should be of poly-tetrafluoroethylene (PTFE) or similar inert material.

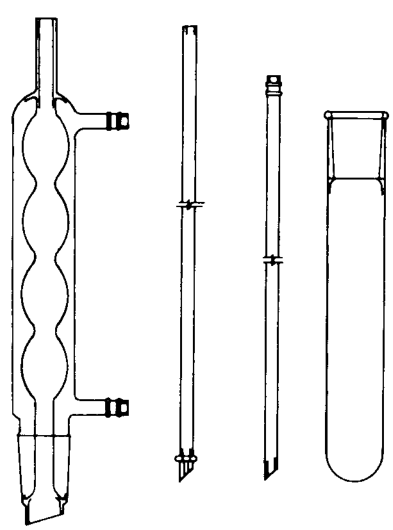

FIG. 2 Apparatus

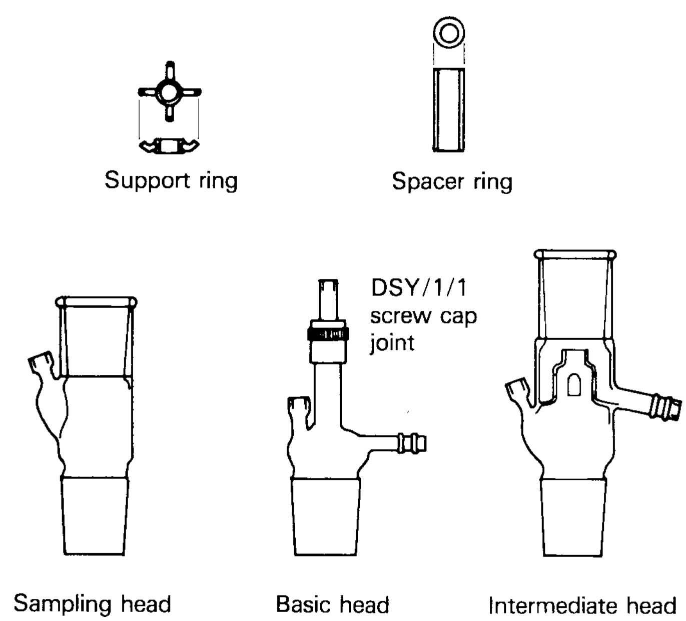

FIG. 3 Apparatus

high-mounted glass ring. Four glass hooks are spaced at 90° intervals from which catalyst coupons may be suspended. See Fig. 3.

5.5.9 *Spacer Ring*, 9.5-mm inside diameter, 12.7-mm outside diameter, 7 mm long, to fit on inlet tube I fitted with low-mounted glass ring. The spacer is added to separate metal washers used as catalyst and corrosion test specimens. See Fig. 3.

6. Procedure

6.1 *Test Conditions*:

6.1.1 Stability tests can be run for a fixed time period, with measurement of selected parameters at the end of the test period or at intervals throughout the test.

6.1.2 Stability tests can be run until a predetermined failure point, with measurement of selected parameters to determine failure. Samples of fluid are withdrawn at intervals to measure changes in the parameters.

6.1.3 Fixed time tests are convenient for setting performance specifications; variable time tests can offer more useful information in screening tests and evaluating formulation changes.

6.2 Possible test parameters that can be monitored include:

6.2.1 Change in acid number (Test Methods D 664, D 974, D 3339, or D 5770),

6.2.2 Change in precipitation number (Test Method D 91),

6.2.3 Change in viscosity (Test Method D 445),

6.2.4 Change in color of fluid (Test Methods D 156 or D 1500),

6.2.5 Evaporation loss from test fluid,

6.2.6 Formation of an insoluble sludge,

6.2.7 Formation of a spot on a test blotter,

6.2.8 Formation of varnish,

6.2.9 Change in weight of catalyst specimens,

6.2.10 Change in appearance of catalyst specimens,

6.2.11 Weight of material volatilized from sample, and

6.2.12 Acid number of volatilized material (Test Method D 3339 or D 5770).

7. Keywords

7.1 accelerated testing-petroleum products; gear oil-stability test apparatus; hydraulic oil-stability test apparatus; oxidation testing-petroleum; stability-oxidation; stability-thermal; turbine oil-stability test apparatus; universal oxidation test apparatus