Designation: D7098 – 08$^{\varepsilon 1}$

Standard Test Method for Oxidation Stability of Lubricants by Thin-Film Oxygen Uptake (TFOUT) Catalyst B[1, 2]

This standard is issued under the fixed designation D7098; 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.

ε^1 NOTE—Updated units statement in 1.3 and improved figure quality editorially in November 2009.

1. Scope*

1.1 This test method covers the oxidation stability of lubricants by thin-film oxygen uptake (TFOUT) Catalyst B. This test method evaluates the oxidation stability of petroleum products, and it was originally developed as a screening test to indicate whether a given re-refined base stock could be formulated for use as automotive engine oil[3] (see Test Method D4742). The test is run at 160°C in a pressure vessel under oxygen pressure, and the sample contains a metal catalyst package, a fuel catalyst, and water to partially simulate oil conditions in an operating engine. In addition, the test method has since been found broadly useful as an oxidation test of petroleum products.[4]

1.2 The applicable range of the induction time is from a few minutes up to several hundred minutes or more. However, the range of induction times used for developing the precision statements in this test method was from 40 to 280 min.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3.1 *Exception*—Pressure units are provided in psig, and dimensions are provided in inches in Annex A1 and Annex A2, because these are the industry accepted standard and the apparatus is built according to the figures shown.

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:*[5]

A314 Specification for Stainless Steel Billets and Bars for Forging

B211 Specification for Aluminum and Aluminum-Alloy Bar, Rod, and Wire

D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration

D1193 Specification for Reagent Water

D2272 Test Method for Oxidation Stability of Steam Turbine Oils by Rotating Pressure Vessel

D4742 Test Method for Oxidation Stability of Gasoline Automotive Engine Oils by Thin-Film Oxygen Uptake (TFOUT)

E1 Specification for ASTM Liquid-in-Glass Thermometers

E144 Practice for Safe Use of Oxygen Combustion Bombs

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *break point*—the precise point of time at which rapid oxidation of the oil begins.

3.1.2 *oxidation induction time*—the time until the oil begins to oxidize at a relatively rapid rate as indicated by the decrease of oxygen pressure.

3.1.3 *oxygen uptake*—oxygen absorbed by oil as a result of oil oxidation.

4. Summary of Test Method

4.1 The test oil is mixed in a glass container with four other liquids used to simulate engine conditions: (*1*) an oxidized/nitrated fuel component (Annex A3), (*2*) a mixture of soluble

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.09.0G on Oxidation Testing of Engine Oils.

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[2] While Catalyst B can be used for testing oxidation stability of many lubricant types, the mixture of fuel, nitro-paraffin, and catalyst components used in this test method simulates the Sequence IIIE Engine Test. Test results on several ASTM reference oils have been found to correlate with Sequence IIIE engine tests in hours for a 375 % viscosity increase. (See Ku, Chia-Soon, Pei, Patrick T., and Hsu, Stephen M., "A Modified Thin-Film Oxygen Uptake Test (TFOUT) for the Evaluation of Lubricant Stability in ASTM Sequence IIIE Test, SAE Technical Paper Series 902121, Tulsa, OK, Oct. 22-25, 1990.)

[3] Ku, C. S. and Hsu, S. M., "A Thin Film Uptake Test for the Evaluation of Automotive Lubricants," *Lubrication Engineering*, 40, 2, 1984, pp. 75–83.

[4] Selby, Theodore W., "Oxidation Studies with a Modified Thin-Film Oxygen Uptake Test", *SAE Technical Paper Series* 872127, Toronto, Ontario, Nov. 2-5, 1987.

[5] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

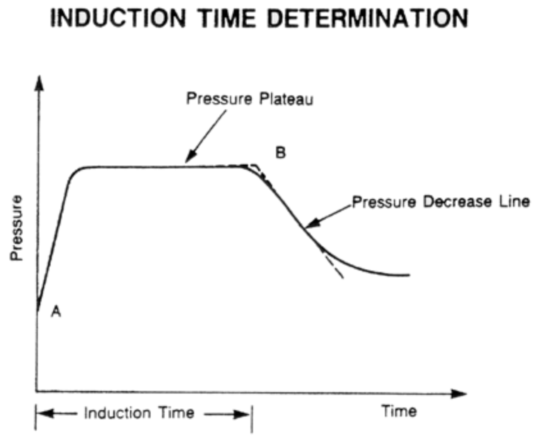

FIG. 1 Pressure versus Time Diagram of the Oxidation Test

metal naphthenates (lead, iron, manganese, and tin naphthenates (Annex A4), (*3*) a nitro-paraffinic compound, and (*4*) Type I reagent water.

4.2 The glass container holding the oil mixture is placed in a pressure vessel equipped with a pressure sensor. The pressure vessel is sealed, charged with oxygen to a pressure of 620 kPa (90 psig), and placed in an oil bath at 160°C at an angle of 30° from the horizontal. The pressure vessel is rotated axially at a speed of 100 r/min forming a thin film of oil within the glass container resulting in a relatively large oil-oxygen contact area.

4.3 The pressure of the pressure vessel is recorded continuously from the beginning of the test and the test is terminated when a rapid decrease of the pressure vessel pressure is observed (Point B, Fig. 1). The period of time that elapses between the time when the pressure vessel is placed in the oil bath and the time at which the pressure begins to decrease rapidly is called the oxidation induction time and is used as a measure of the relative oil oxidation stability.

5. Significance and Use

5.1 This test method was originally developed to evaluate oxidation stability of lubricating base oils combined with additives chemistries similar to those found in gasoline engine oils and service.[2]

5.2 This test method is useful for screening formulated oils before engine tests. Within similar additive chemistries and base oil types, the ranking of oils in this test appears to be predictive of ranking in certain engine tests. When oils having different additive chemistries or base oil type are compared, results may or may not reflect results in engine tests. Only gasoline engine oils were used in generating the precision statements in this test method.

6. Apparatus

6.1 *Oxidation Bath and Pressure Vessel*—See appropriate Annex (Annex A1[6] or Annex A2[7]) for detailed description of apparatus and accessories for equipment described in this test method.

NOTE 1—To reduce vapor odors when opening pressure vessel after use, a hood may be desirable.

6.2 *Precision Pressure Gauge*—Use a certified precision pressure gauge to accurately control the oxygen feed to the pressure vessel. The gauge shall have a sufficient range to encompass 0 to 650 kPa (~90 psig) required by the test method with division 2.0 kPa (~0.5 psig) or better to enable readings to be made to 2.0 kPa (~0.25 psig).

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.[8]

7.2 *Purity of Water*—Unless otherwise indicated, references to reagent water shall be understood to mean distilled water meeting requirements of reagent water as defined by Type I of Specification D1193.

7.3 *Acetone*, CH_3COCH_3.

7.4 *Air*, containing 2000 ppm nitrogen dioxide, NO_2 (commercially available compressed gas mixture, certified within ±5 %).

7.5 *Cyclo-hexane*, C_6H_{12}, Practical Grade or other suitable hydrocarbon solvent. (**Warning**—Highly flammable. Skin irritant on repeated contact. Aspiration hazard.)

7.6 *Isopropyl Alcohol*, $CH_3CH(CH_3)OH$.

7.7 *Oxygen*, 99.8 %.

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[6] The sole source of supply of the apparatus known to the committee at this time is Koehler Instrument Co., Inc., 1595 Sycamore Ave., Bohemia, NY11716 and Stanhope-Seta, London St., Chertsey, Surrey, KT16 8AP, U.K. 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.

[7] The sole source of supply of the apparatus known to the committee at this time is Tannas Co., 4800 James Savage Rd., Midland, MI 48642. 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.

[8] *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 *Annual 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.

8. Materials

8.1 *TFOUT Catalyst B Package:*[7]

8.1.1 *Fuel Component*—The fuel component is a nitrated gasoline fraction or organic equivalent. This component may be prepared in accordance with the procedures described in Annex A3.

8.1.2 *Soluble Metal Catalyst Mixture*—This catalyst is a mixture of soluble metal catalysts (lead, iron, manganese, and tin). The catalyst may be prepared according to the procedures described in Annex A4.

8.1.2.1 Other oxidation stability test methods have demonstrated that soluble metal catalyst supplies may be inconsistent and have significant effects on the test results. Thus, for test comparisons, the same source and same batch of metal naphthenates shall be used.

NOTE 2—It is good research practice to use the same batches of catalyst components when closely comparing engine oils.

NOTE 3—Slow, steady reactivity of some of the catalyst chemicals can be a problem. Such problems can be reduced by storing the closed catalyst vials in a refrigerator at approximately 5°C. The catalyst chemicals remain effective up to six months after the septum is punctured, if they are stored as noted above.

8.1.3 *Nitro-paraffin*—This compound is made up of a nitrialkane blend.

NOTE 4—Suitably prepared catalyst packages may be purchased from Tannas Co.[7]

8.2 *Varnish and Deposit Remover*, water-soluble varnish remover or other engine varnish/deposit removers.

8.3 *Silicone Stopcock Grease*.

9. Preparation of Apparatus

9.1 *Glass Sample Container*—A clean glass sample container is important for obtaining repeatable results. Thorough cleaning can be accomplished by (*a*) rinsing with cyclo-hexane or other suitable hydrocarbon solvent, (*b*) soaking in concentrated solution of a water-soluble varnish remover, (*c*) thoroughly rinsing with water, (*d*) rinsing with acetone, (*e*) and permitting to dry.

NOTE 5—A segmented glass reaction dish has been found suitable to prevent premature mixing of the catalyst components (see Fig. A2.4)

9.2 *Cleaning of Pressure Vessel*—Fill with concentrated solution of a water-soluble varnish remover and soak for suitable time, rinse with water, rinse with acetone, and permit to dry.

9.3 *Cleaning of Pressure Vessel Stem*—Periodically disassemble, inspect, and clean the pressure vessel stem. Rinse the inside of the stem with isopropyl alcohol and blow dry with oil free compressed air. For users of apparatus described in Annex A1, periodically insert a dry pipe cleaner into the transducer line opening for removal of potential residue buildup.

NOTE 6—Replace O-rings when reassembling the pressure transducers.

9.4 Periodically pressure test the pressure vessels at 690 kPa (~100 psi) with air or oxygen. If the pressure drops more than 0.690 kPa (~0.1 psi) on the pressure gauge within 60 s, replace the O-ring seals and inspect the valve seals according to manufacturer's directions. If the problem continues, contact the specific equipment manufacturer.

NOTE 7—Previous versions of this test method have called for hydrostatic testing of the pressure vessel. This was found unnecessary at the relatively low pressures involved in running this test method.

9.5 *Cleaning of Catalyst Syringes*—Use individual catalyst syringes for each catalyst component. Thoroughly clean and dry syringes prior to each use. (See Annex A5 for recommended procedure.)

10. Procedure

10.1 *Weighing and Mixing Sample and Catalyst Components*:

10.1.1 Place the clean glass sample container onto the precision balance and tare.

10.1.2 Weigh 1.500 ± 0.001 g of oil sample into the container and tare.

10.1.3 Add 0.045 ± 0.001 g of the soluble metal catalyst mixture into the glass sample container and tare.

10.1.4 Add 0.030 ± 0.001 g each of the fuel component, nitro-paraffin and reagent water to the glass sample container and tare each time. It is easiest to add the distilled water last and place on top of the oil sample.

10.1.5 Just prior to inserting the glass sample container into the pressure vessel, thoroughly mix the catalyst components within the sample container by hand-rotation (approximately five rotations) and proceed immediately to 10.2. Delay may result in variation of results.

10.2 *Pressure Vessel Assembly and Charging*—Immediately and rapidly assemble and charge the pressure vessel in accordance with apparatus type (see A1.2 or A2.7).

NOTE 8—Avoid releasing the oxygen too rapidly by decreasing the pressure to atmospheric in no less than 1 min to avoid possible foaming and overflow of the sample from the glass sample container.

10.3 *Oxidation*—Before starting the test, bring the heating bath to the test temperature at 160°C and insert the pressure vessel(s) in accordance with apparatus type (see A1.3 or A2.8).

10.3.1 Allow the bath temperature to level out at the test temperature, which must occur within 15 min after insertion of the pressure vessel. Maintaining the test temperature within the specified limits of 160 ± 0.3°C during the entire test run is the most important single factor ensuring both repeatability and reproducibility of test results. If the test temperature cannot be maintained as specified, the test results shall not be considered valid.

NOTE 9—The time for the bath to reach the operating temperature after insertion of the pressure vessel may differ for different apparatus assemblies and shall be observed for each unit (a unit may carry one, two, three, or four pressure vessels). The objective is to find a set of conditions, which does not permit a drop of more than 2°C after insertion of the pressure vessel(s) and allows the pressure vessel pressure to reach plateau within 15 min.

10.4 Keep the pressure vessel completely submerged and maintain continuous and uniform rotation throughout the test. A standard rotational speed of 100 ± 5 r/min is required; any variation in this speed could cause erratic results.

10.5 Monitor the pressure of the pressure vessel preferably using a strip chart or some other form of electronic data collection program. If a dial pressure gauge is used, make readings at least every 5 min. (The maximum pressure must be reached within 15 min.) After a test period (the induction time), the pressure decreases because of oxygen absorption by oil (the break point).

10.5.1 When the oil reaches the break point, the pressure decreases rapidly as oxygen is absorbed rapidly by the test oil. The test can be terminated as soon as sufficient information has been collected to form a tangent to the decreasing pressure trace (see 10.6) or, if desired, continued until pressure decreases to some further level.

NOTE 10—The pressure within the pressure vessel increases at the beginning because of gas expansion accompanying the temperature increase of the pressure vessel. Following this rise, the pressure reaches a plateau as shown in Fig. 1. This pressure may gradually drop slightly during the test. A gradual decrease of the pressure is not unusual and does not invalidate the test. The time between initiating the test and the break point is called the oxidation induction time.

NOTE 11—If a break in pressure does not occur within 300 to 500 min, the operator may elect to terminate the test. A slow decrease in pressure may also indicate a small leak from the pressure vessel, which is why it is a good practice to occasionally determine whether a slow leak is present.

10.6 Record the time at which the pressure starts to decrease rapidly at the break point (Point B, Fig. 1), which is marked as the intersection of the tangent of the pressure plateau line during the final 20 min before the break point and the tangent of the pressure decrease line following the break point as shown in Fig. 1.

11. Report

11.1 Report the oxidation induction time in minutes. Determine the induction time as the time period from the beginning of the test (Point A, Fig. 1) to the break point (Point B, Fig. 1).

12. Precision and Bias [9]

12.1 The precision of this test method, as determined by statistical examination of interlaboratory results on break point time, is as follows:

12.1.1 *Repeatability*—The difference between successive 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:

14 % of mean

12.1.2 *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:

39 % of mean

12.2 The range of induction times used for developing this precision statement was from 40 to 280 min.

12.3 *Bias*—No information can be presented on the bias of the procedure in this test method for measuring oxidation stability because no material having an accepted reference value is available.

12.4 The precision statements in 12.1.1 and 12.1.2 were determined from an interlaboratory study using the same batch of soluble metal mixture (TFOUT Catalyst B Package of Tannas Co.).

13. Keywords

13.1 oxidation stability; sequence IIIE engine simulation; TFOUT

ANNEXES

(Mandatory Information)

A1. THIN FILM OXYGEN UPTAKE TEST USING THE RBOT/TFOUT APPARATUS

INTRODUCTION

Two types of TFOUT instruments were used in generating the precision data given in this test method. The first was the modified RBOT (now known as RPVOT) instrument originally used to develop the test procedure and for distinction is called the RBOT/TFOUT apparatus. The second was an instrument designed specifically to run the TFOUT test and later modified to permit running the RPVOT test.

NOTE A1.1—This annex utilizes two modified RPVOT (Test Method D2272) apparatus of similar design for running the TFOUT test. However, strain-gauge pressure transducers and a computer were incorporated into the later version.

A1.1 *Pressure Vessel*, with lid, cap, and stem is constructed as shown in Fig. A1.1. The pressure vessel has the same dimensional specifications as the RPVOT pressure vessel (see

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[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1571, including the raw data and the statistical treatment of data.

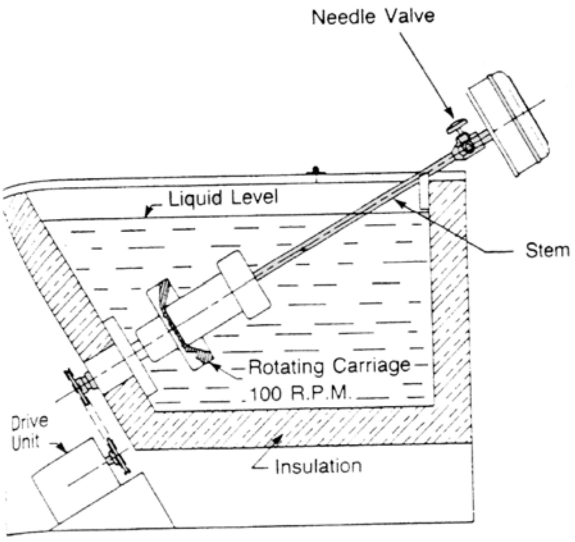


FIG. A1.1 Schematic Drawing of Oxidation Test Apparatus

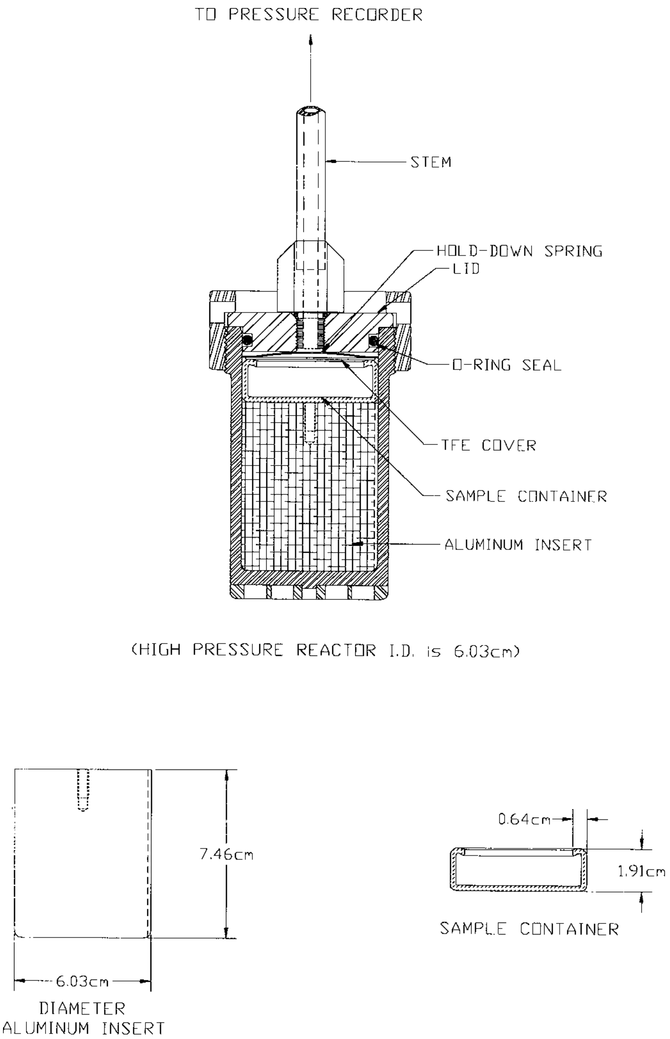
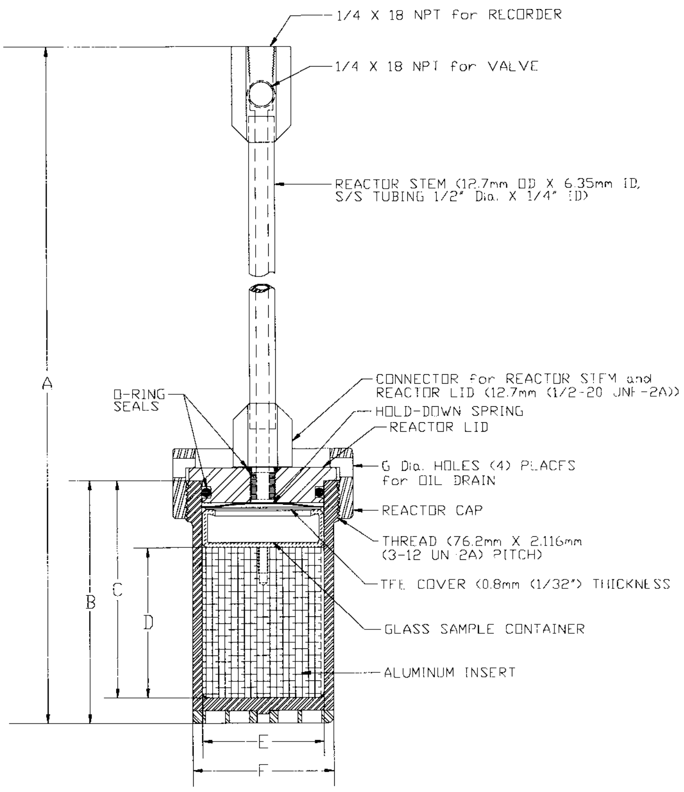


FIG. A1.2 Schematic Drawing of an Assembled Vessel, Aluminum Insert, and Glass Sample Container



DIMENSIONS

	mm	in.
A	536	21.110
B	120.6	4.750
C	107.9	4.250
D	74.6	2.937
E	ø60.3/60.6	ø2.375 +.010/-.000
F	ø69.8	ø2.750
G	ø9.5	ø.375

FIG. A1.3 Construction of Oxidation Vessel

Test Method D2272). Therefore, the pressure vessel for RPVOT can be used for this test. However, in the test an aluminum insert and a glass sample container, as specified in A1.4 and A1.5, respectively, are to be used.[3]

A1.1.1 *Pressure Vessel Body and Lid*, are to be made of 303 stainless steel (see Specification A314). The pressure vessel body is to be machined from 76.2-mm (3.00-in.) solid stainless steel. The interior surface shall be given a smooth finish to facilitate cleaning.

A1.1.2 *Pressure Vessel Stem*, is to be constructed of 303 stainless steel, the stem having an inside diameter of 6.4-mm (0.25-in.) and is to be equipped with a 6.4-mm needle valve. The stem is connected to the center of the lid as shown in Fig. A1.3.

A1.1.3 *Pressure Vessel Cap (or Closure Ring)*, is to be made of chrome-plated steel.

A1.1.4 *O-ring Gaskets*, silicone or a fluorinated elastomer, 50.8-mm (2.00-in.) inside diameter by 60.3-mm (2.375-in.) outside diameter.

A1.2 *Pressure Vessel Assembly and Charging*—After proper cleaning (see Section 9), assemble the pressure vessel as shown in Fig. A1.2.

A1.2.1 Put the aluminum insert into the pressure vessel followed by the glass sample container and the TFE (tetrafluoro ethylene) plastic cover disk.

A1.2.2 Place the stainless hold-down spring on top of the TFE disk and the glass sample container.

Note A1.2—The stainless steel hold-down spring not only holds down the TFE cover disk but also, more importantly, prevents the glass sample container from slipping inside the pressure vessel with consequent poor results.

A1.2.3 Apply a thin coating of silicone stopcock grease to the O-ring high pressure vessel seal located in the gasket groove of the pressure vessel lid to provide lubrication.

A1.2.4 Insert the lid into the pressure vessel.

A1.2.5 Place and tighten the cap on the pressure vessel to lock down the lid in the pressure vessel.

A1.2.6 Attach the pressure oxygen hose and purge the pressure vessel twice at about 620 kPa (90 psig) of oxygen to remove the air originally present in the pressure vessel. (**Warning**—See Note 8.)

A1.2.7 Charge the pressure vessel to 620 kPa (90 psig) oxygen at 21°C using an in-line precision pressure gauge to monitor the pressure vessel pressure with 1 kPa (0.12 psig). (For ambient temperatures other than 21°C, increase (or decrease) the initial pressure by 2.5 kPa (0.4 psig) for each 1°C above (or below) 21°C.)

A1.2.8 Fill the pressure vessel to the required pressure and close the inlet valve. Test the pressure vessel for leaks by immersing in water or by using soap solution.

A1.2.9 Proceed to 10.3.

A1.3 *Oxidation*—Before starting the test, bring the heating bath to the test temperature of 160°C while the stirrer is in operation. (This can be done during preparation of the sample and the pressure vessel.)

A1.3.1 Switch off the stirrer and insert the pressure vessel into the carriages. Note the time, and restart the stirrer.

A1.3.2 If an auxiliary heater is used, keep it on for the first 5 min of the run and then turn it off (Note 9).

A1.3.3 Proceed to 10.3.1.

A1.4 *Aluminum Insert*, (Fig. A1.2) made of 2024 aluminum rod (see Specification B211) with 60.3 mm in diameter and 74.6 mm in height.

A1.5 *Glass Sample Container*, (Fig. A1.2) constructed of borosilicate glass.

A1.5.1 The top of the sample container is to be covered with a 57.2 mm (2.25 in.) diameter TFE-fluorocarbon disk with a 3.2 mm (0.125 in.) diameter hole in the center. The TFE cover prevents sample splash during vessel charge. The disk shall have a thickness of 0.8 mm (0.03149 in.). As an added safeguard against the occurrence of rotational differences between the glass sample container and pressure vessel, a hold-down spring is required.

A1.5.2 The glass sample container shall have a sliding fit in the pressure vessel with no excess side clearance. The container alone is to have a maximum wall thickness of 2.5 mm.

A1.6 *Gauge*, recording, as shown in Fig. A1.4, or indicating, with a range from 0 to 1400 kPa (~200 psi) and graduated in 25 kPa (or 5 psi) divisions.

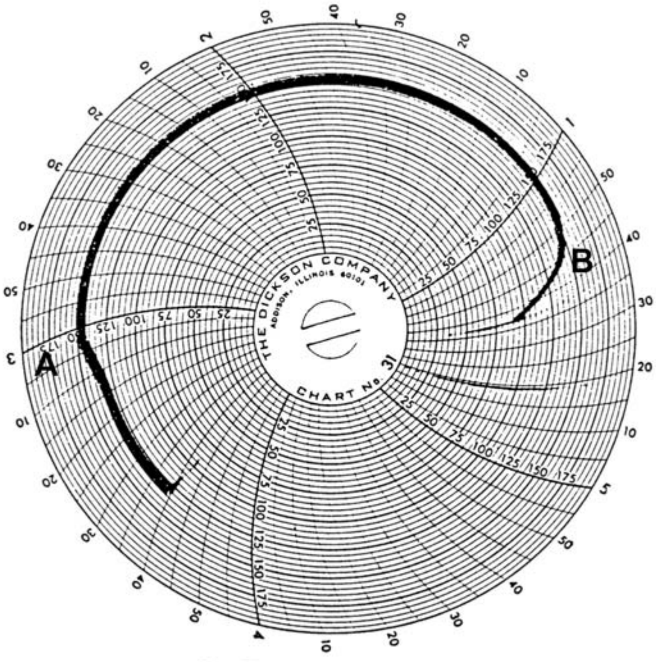


FIG. A1.4 Chart of Recording Pressure Gauge (Actual Size—144 mm (4½ in.))

A1.6.1 The accuracy must be 2 % or less of the total scale interval.

A1.6.2 Mount recording gauges so that their faces are perpendicular to the axis of rotation.

Note A1.3—Clock-wound, round chart recorders shall be periodically checked for constancy of the clock works and agreement with standard time.

A1.7 *Oxidation Bath*, equipped with an efficient stirrer and with a suitable device for holding and rotating the pressure vessel axially at an angle of 30° at 100 ± 5 r/min while submerged in oil to a point at least 25.4 mm (1 in.) below the level of the bath liquid (Note 2). Keep the oil level at 50.8-mm (2-in.) below the bath cover.

A1.7.1 A bath at least 229-mm (9-in.) deep, filled with 30 L (8 gal) of heavy bath oil per pressure vessel (petroleum or synthetic oil having a flash point greater than 250°C). Metal block baths are not satisfactory for this service.

A1.7.2 Provide thermal regulation to maintain the bath within ±0.3°C of the test temperature for a period as long as 8 h. There shall be sufficient heat immediately available to bring the high pressure vessels to operating temperature of 160°C within 15 min.

A1.8 *Thermometer*, digital or liquid-in-glass styles shall be used to check the bath temperature monthly (or more often, if necessary). The thermometer shall be able to be read with an accuracy of ±0.1°C at the level of 160°C. Thermometers, digital or analog, need to be checked for accuracy at least one time per year. If using the ASTM Solvents Distillation 102C-86 thermometer, then use as prescribed in Specification E1.

A2. THIN FILM OXYGEN UPTAKE TEST USING THE DESIGNED TFOUT APPARATUS

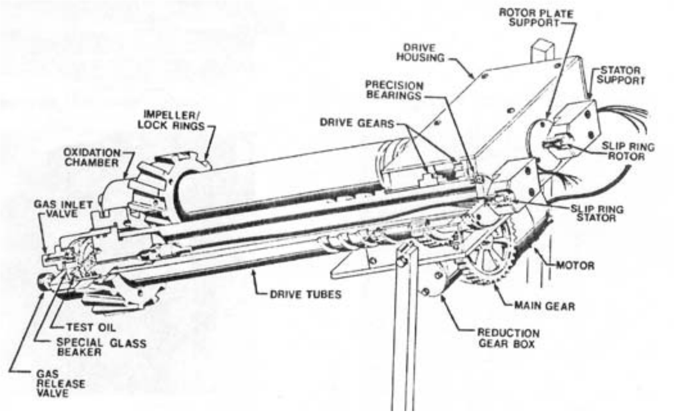


FIG. A2.1 Construction of Oxidation Vessel

A2.1 *Pressure Oxidation Vessel*, for the TFOUT apparatus is constructed as shown in Fig. A2.1. The TFOUT apparatus is designed to utilize the more sensitive strain-gauge technology and electronic amplification with a linear chart recorder or computer.

A2.2 *Oxidation Chamber*, is comprised of a reaction vessel cover and body constructed of highly polished 304 stainless steel.

A2.2.1 *Reaction Vessel Body*, (Fig. A2.2), houses the reaction vessel cover and a special glass reaction dish and attaches to the drive tube. The vessel body measures 69.9-mm (2.75-in.) in diameter and 63.5-mm (2.5-in.) in length, with fittings for the gas inlet and gas outlet valves.

A2.2.2 *Reaction Vessel Cover*, (Fig. A2.3), protrudes from the reaction vessel body into the drive tube, with a 1/4-18 NPT drilled and tapped hole for connection with the pressure transducer fitting. Two O-rings, as specified in A2.5, are used for a pressure seal.

A2.3 *Drive Tube*, (Fig. A2.3), is to be constructed of 304 stainless steel, polished to a mirror-like finish, having an overall length of 493.3-mm (19.5-in.) and inside diameter of 39.7-mm (1.563-in.).

A2.4 *Impeller/Lock Ring*, (Fig. A2.3), is constructed of a highly polished 304 stainless steel with an outside diameter of 114.3 mm (4.5 in.) and thickness of 44.5-mm (1.75 in.), equipped with nine 30° angle grooves utilized for stirring once the reactor is lowered into the bath. The impeller/lock ring fastens the reaction vessel body to the drive tube.

A2.5 *O-ring Gaskets*, silicone or a fluorinated elastomer, deterioration occurs over time.

A2.5.1 *Reaction Vessel O-ring*—Use two per reaction vessel cover, change after 50 runs (OD = 59.26-mm (2.33-in.)).

A2.5.2 *Gas Inlet O-ring*—Use one per gas inlet valve, change after two runs (OD = 14.29-mm (0.56-in.)).

A2.5.3 *Dual Valve O-ring*—Use one per gas inlet valve, change after 20 runs, and two per gas outlet valve, change after 50 runs (OD = 7.94-mm (0.31-in.)).

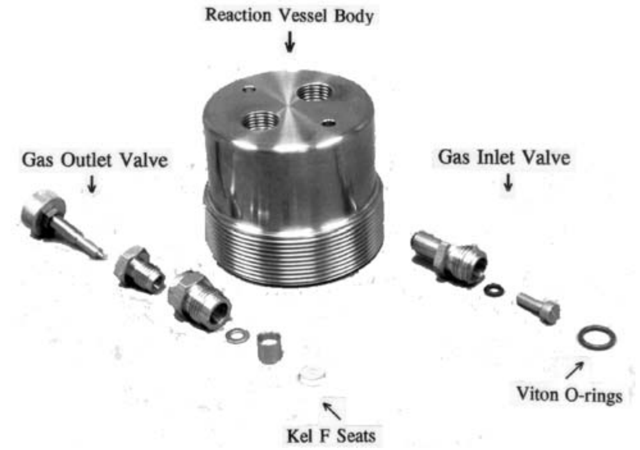


FIG. A2.2 Photo of Reaction Vessel Body Assembly

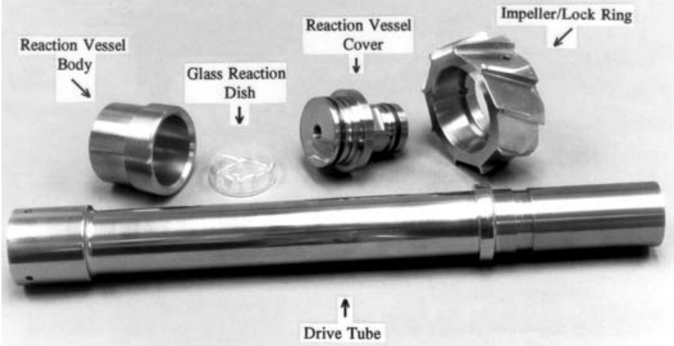


FIG. A2.3 Photo of High Pressure Vessel Components

A2.6 *Glass Reaction Dish*, (Fig. A2.4), is a specially segmented glass beaker constructed of borosilicate glass with an outside diameter of 60-mm (2.36-in.).

A2.7 *Pressure Vessel Assembly and Charging*—After proper cleaning (see Section 9), assemble the high pressure vessel as shown in Fig. A2.1.

A2.7.1 Assemble the gas inlet and gas outlet valves on reaction vessel body according to Fig. A2.2.

A2.7.2 Insert the segmented glass reaction dish containing the catalyst and sample mixture (10.1.5), being careful not to overturn reaction dish, into the vessel body.

A2.7.3 Carefully slide the vessel body over the reaction vessel cover and secure to drive tube by tightening the impeller/lock ring.

A2.7.4 Attach the pressure oxygen hose and purge the pressure vessel twice at about 620 kPa (90 psig) of oxygen to remove the air originally present in the pressure vessel. (**Warning**—See Note 8.)

A2.7.5 Charge the pressure vessel to 620 kPa (90 psig) oxygen at 21°C using an in-line precision pressure gauge to monitor the pressure vessel pressure with 1 kPa (0.12 psig). (For ambient temperatures other than 21°C, increase (or decrease) the initial pressure by 2.5 kPa (0.4 psig) for each 1°C above (or below) 21°C. (See Practice E144.)

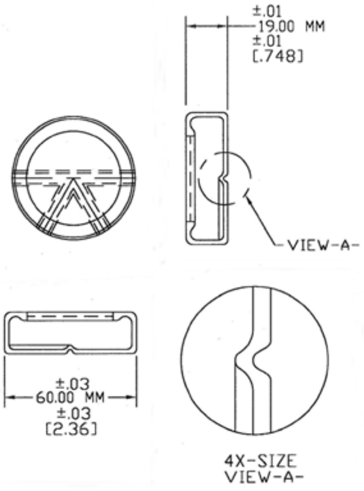


FIG. A2.4 Drawing of Segmented Glass Reaction Dish

A2.7.6 Tighten the inlet valve and carefully lower the pressure vessel into the heating bath.

A2.7.7 Proceed to 10.3.

A2.8 *Oxidation*, Faster rise time to operating temperature is additionally enhanced by bringing the bath to 175°C before immersion of the pressure vessels. Immediately following immersion of the vessels, set the bath temperature to control level of 160°C. This technique produces a more repeatable and rapid test, allowing the vessel pressure to reach a plateau within 15 min.

A2.8.1 Proceed to 10.3.1.

A2.9 *Oxidation Bath*, is a 14.7 L (3.25 gal) stainless steel bath filled with heavy bath oil (petroleum or synthetic oil having a flash point greater than 225°C). Metal block baths are not satisfactory for this service. Follow the manufacturer's guidelines on filling the bath level.

A2.10 Provide thermal regulation to maintain the bath within ±0.3°C of the test temperature.

A2.11 *Recorder*—A linear three-pen chart recorder analysis (Fig. 1), is used to record pressure vessel pressure charge and temperature.

A2.12 *Thermometer*, see A1.8.

A3. PREPARATION OF THE FUEL COMPONENT

A3.1 This annex describes the experimental procedures and the materials for preparation of the fuel component for the thin-film oxygen uptake. The fuel component is an oxidized/nitrated high boiling gasoline fraction.

A3.2 Prepare the fuel component in accordance with the following procedures: (**Warning**—The fuel component is dangerous when exposed to heat or flame. It can also react vigorously with oxidized materials. Use a well-ventilated laboratory for each of the steps.)

A3.3 *Distillation*—Use ASTM certified V-D engine test fuel as the material. A typical V-D fuel contains 40 volume % aromatics, 12 volume % olefins, and 43 volume % paraffins and naphthenates. The boiling range is from 34 to 209°C, and the density is 0.76 g/mL. Obtain the fuel from the supplier given in ASTM STP 315H.[10] Obtain a high-boiling fraction (>150°C (normal boiling point)) as residue by vacuum distillation (that is, 5 cm Hg) of the V-D fuel. (Commercially available glass distillation apparatus with a separable distilling head is found satisfactory for this purpose.)

A3.4 *Oxidation/Nitration*—The oxidation apparatus is shown in Fig. A3.1. Oxidize 100 g of the high boiling fraction at 125°C by bubbling air through the fuel at 100 mL /min. The air contains 2000 mg/kg nitrogen dioxide. During oxidation, permit volatile oxidation products to escape to ventilation. The high-boiling oxidized/nitrated fuel component remains in the oxidation tube. Periodically withdraw a small sample and test it for the acid number (see Test Method D664). Terminate the oxidation when the acid number of the reaction product reaches between 10 to 15 mg of KOH/g.

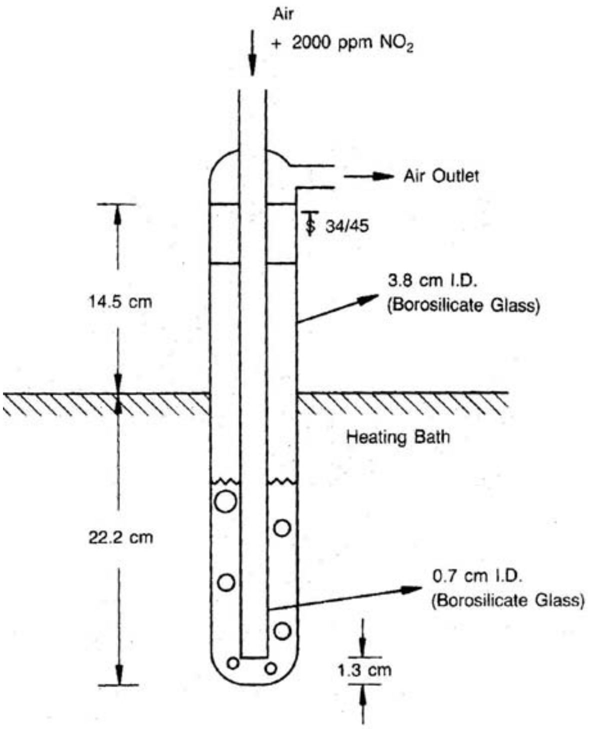


FIG. A3.1 Oxidation Apparatus for Fuel Fraction

A3.5 *Reduction of Acidity*—Reduce the acidity of the reaction product for A3.4 with a saturated solution of reagent grade (99.9 %) sodium bicarbonate (in Type II reagent water). Place

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[10] "Part 3: Sequence V-D," *Multicylinder Test Sequences for Evaluating Automotive Engine Oils, ASTM STP 315*, ASTM International.

the reaction product in a 500 mL separatory funnel, and shake it with equal quantity of the sodium bicarbonate solution. When completed, the reaction product should have an acid number of 2 ± 0.1. After the sodium bicarbonate solution is drained from the separatory funnel, collect and use the reacted product as the fuel components.

A3.6 In summary, the fuel component is prepared through oxidation/nitration of a high boiling fraction of ASTM V-D engine test fuel. Control the reactivity of the fuel component by neutralization of the oxidation/nitrated fuel component to an acid number of 2.

A3.7 There shall be no change to the composition of the fuel. However, new batches must give oxidation results within the precision of the method using at least one reference oil having prior well-established thin film oxygen uptake test (TFOUT) values.

A4. PREPARATION OF THE SOLUBLE METAL CATALYST MIXTURE

A4.1 This annex describes the materials and the experimental procedures for preparation of the soluble metal catalyst mixture for the thin film oxygen uptake test (TFOUT). The metal mixture contains five metal naphthenates including lead, ferric, cupric, manganese, and stannous naphthenates.

A4.2 *Materials*—Commercially available naphthenates in mineral spirits are used. The materials used are lead naphthenate, ferric naphthenate, manganese naphthenate, and stannous naphthenate. When purchased, lead naphthenate contains approximately 24 wt-% of lead, stannous naphthenate approximately 16 wt-% as tin, and other naphthenate approximately 6 wt-% as the respective metal. Filter these materials (0.2 mL TFE filter) under a pressurized filtration system to remove any particulate substances.

A4.3 *Metal Content*—Determine the metal content within ±5 % of the mean of each naphthenate by atomic absorption spectroscopy or other equivalent method.

A4.4 *Mixing of Metal Naphthenates*—Prepare the soluble metal catalyst mixture by mixing naphthenate components in the proportion so that the metal ratio in the mixture is as specified in A4.5. These components are stirred vigorously to achieve uniform mixing (for example, 100 g of material in a 250 cm^3 container should be stirred for 2 h at medium speed using a 2.5-cm magnet).

A4.5 *Composition of the Metals*—The concentration of the total metal content in the mixture should be 0.18 ± 0.02 g of metal/g of mixture. The mixture contains 14.7 ± 0.5 wt-% of lead metal, 0.75 ± 0.01 wt-% of iron metal, 0.65 ± 0.01 wt-% of manganese metal, and 0.67 ± 0.01 wt-% of tin metal. The ratio of the metal element in the mixture then is approximately as follows:

Metal	Metal Ratio (Weight % of Total Metal Content)
Lead	81.50
Iron	4.2
Manganese	3.6
Tin	3.7

A4.5.1 There shall be no change to the composition of the metal catalyst. However, new batches must give oxidation results within the precision of the method using at least one reference oil having prior well-established thin film oxygen uptake test (TFOUT) values.

A4.6 In summary, prepare the soluble metal mixture from pre-filtered commercially available metal naphthenates.[11] Determine metal content in each naphthenate by atomic absorption spectroscopy. The metal mixture has a composition as specified in A4.5.

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[11] The mixture was prepared using metal naphthenates from the specified batch number of the supplier: lead naphthenate (No. 101882 of Pfaltz & Bauer, Inc., 172 E. Aurora St., Waterbury, CT 06708), ferric naphthenate (No. 7124 of Pfaltz & Bauer, Inc.), manganese naphthenate (No. 25296 of Pfaltz & Bauer, Inc.), and stannous naphthenate (No. 32519-A of K&K Division, ICN Pharmaceuticals, Inc. (Valeant Pharmaceuticals Inc.), 3300 Hyland Ave., Costa Mesa, CA 92626.). 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.

A5. CLEANING PROCEDURE FOR CATALYST SYRINGES

A5.1 This annex describes the materials and recommended procedure for proper cleaning of glass barrel micro-syringes used to load the catalyst components and oil sample into the glass sample container. Glass barrel micro-syringes shall be cleaned between each use, and it is best to identify each syringe as to the associated liquid component.

A5.2 *Materials*—Commercially available beakers and solvents are found satisfactory for this purpose. The beakers should be approximately 50 mL in size and are used to hold hexane and acetone reagents.

A5.3 Pour approximately 10 mL of hexane or other suitable hydrocarbon solvent and acetone into two separate 50 mL beakers. Completely discharge the 100 mL glass barrel micro-syringes into an appropriate waste liquid container to remove remaining component residue. Withdraw a full syringe of hexane or other suitable hydrocarbon solvent from the beaker of clean solvent, and discharge it into the waste container. Conduct this procedure two more times for a total of three flushes. This same procedure is then immediately followed with the acetone.

A5.4 Immediately after discharging the second and final syringe of acetone into an appropriate waste container, pump the glass barrel micro-syringe plunger several times to dryout the reservoir. When the plunger tends to leak air when filling, remove the plunger and tap the plastic tip onto a hard flat surface.

A5.5 Withdraw the desired amount of component into the appropriately identified clean glass barrel micro-syringe, and inject into the sample container while weighing the injected amount. If the sample container is segmented, slight overfill of a component can be withdrawn and re-injected to more precisely obtain the final weight of that component.

NOTE A5.1—Periodically inspect the needle tip and orifice to determine potential damage and need for replacement.

NOTE A5.2—Replaceable needles of the desired orifice size, designed to fit the glass barrel micro-syringe, are recommended.

SUMMARY OF CHANGES

Subcommittee D02.09 has identified the location of selected changes to this standard since the last issue (D7098–06$^{\varepsilon 1}$) that may impact the use of this standard.

(*1*) Changed Type II to Type I water in 4.1 and 7.2, as was run in the round robin.

(*2*) Modified thermometer selection to include digital style thermometers in A1.8.

(*3*) Added comment to Note 3 regarding catalyst life.

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