



Designation: D525 – 12a

British Standard 4347

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# Standard Test Method for Oxidation Stability of Gasoline (Induction Period Method)<sup>1</sup>

This standard is issued under the fixed designation D525; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the U.S. Department of Defense.*

## 1. Scope\*

1.1 This test method covers the determination of the stability of gasoline in finished form only, under accelerated oxidation conditions. (**Warning**—This test method<sup>2</sup> is not intended for determining the stability of gasoline components, particularly those with a high percentage of low boiling unsaturated compounds, as these may cause explosive conditions within the apparatus. However, because of the unknown nature of certain samples, the pressure vessel assembly shall include a safety burst-disc in order to safeguard the operator.)

NOTE 1—For measurement of oxidation stability of gasoline by measurement of potential gum, refer to Test Method D873, or IP Test Method I38.

NOTE 2—The precision data were developed with gasolines derived from hydrocarbon sources only without oxygenates.

1.2 The accepted SI unit of pressure is the kilo Pascal (kPa), and of temperature is °C.

1.3 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—<http://www.epa.gov/mercury/faq.htm>—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

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 appro-*

*priate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>3</sup>

D873 Test Method for Oxidation Stability of Aviation Fuels (Potential Residue Method)

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

E1 Specification for ASTM Liquid-in-Glass Thermometers

### 2.2 Energy Institute Standards:<sup>4</sup>

IP-138 Test Method for Oxidation Stability, Aviation Gasoline

Part IV—Petroleum and its Products<sup>5</sup>

## 3. Terminology

### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *break point, n*—the point in the pressure-time curve that is preceded by a pressure drop of exactly 14 kPa within 15 min and succeeded by a drop of not less than 14 kPa in 15 min.

3.1.2 *induction period, n*—the time elapsed between the placing of the pressure vessel in the bath and the break point at 100°C.

## 4. Summary of Test Method

4.1 The sample is oxidized in a pressure vessel initially filled at 15 to 25°C with oxygen pressure at 690 to 705 kPa and heated at a temperature between 98 and 102°C. The pressure is recorded continuously or read at stated intervals until the breakpoint is reached. The time required for the sample to reach this point is the observed induction period at the temperature of test, from which the induction period at 100°C

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability and Cleanliness of Liquid Fuels.

This test method has been approved by the sponsoring committees and accepted by the Cooperating Societies in accordance with established procedures.

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<sup>2</sup> Further information can be found in the June 1978, January 1979, and June 1986 editions of the *Institute of Petroleum Review*.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>4</sup> Available from Energy Institute, 61 New Cavendish St., London W1M, 8AR U.K.

<sup>5</sup> A convenient template for measuring the breakpoint is described in the paper by Korb, E. L., "Induction Period Calculator," *ASTM Bulletin*, No. 153, August 1948, pp. 99-102.

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

can be calculated. (**Warning**—In addition to other precautions, to provide protection against the possibility of explosive rupture of the pressure vessel, the pressure vessel should be operated behind an appropriate safety shield.)

## 5. Significance and Use

5.1 The induction period may be used as an indication of the tendency of motor gasoline to form gum in storage. It should be recognized, however, that its correlation with the formation of gum in storage may vary markedly under different storage conditions and with different gasolines.

## 6. Apparatus

6.1 *Oxidation Pressure Vessel, Glass Sample Container and Cover, Accessories, Pressure Gage, and Oxidation Bath*, as described in **Annex A1**.

6.2 *Thermometer*, having a range as shown as follows and conforming to the requirements as prescribed in Specification **E1** or in the specifications for IP thermometers:

NOTE 3—Other temperature sensing devices that cover the temperature range of interest, such as thermocouples or platinum resistance thermometers, that can provide equivalent or better accuracy and precision, may be used in place of thermometers specified in **6.2**.

Temperature Range	Thermometer Number	
	ASTM	IP
95 to 103°C	22C	24C

## 7. Reagents and Materials

7.1 *Gum Solvent*—Mixture of equal volumes of toluene and acetone, both of which shall be of 99 % minimum purity.

7.2 *Oxygen*—Commercially-available extra-dry oxygen of not less than 99.6 % purity.

## 8. Sampling

8.1 Sample in accordance with the procedure for oxidation stability as described in Practice **D4057**.

## 9. Preparation of Apparatus

9.1 Wash the glass sample container with gum solvent until free from gum. Rinse thoroughly with water and immerse the sample container and cover in detergent cleaning solution. The type of detergent and conditions for its use shall match the cleanliness obtained by the use of a strongly oxidizing agent such as chromosulfuric acid, ammonium peroxydisulfate in concentrated sulfuric acid at approximately 8 G./L, or sulfuric acid itself, soaking for at least 12 h, followed by rinses in tap water, distilled water and then acetone. For comparison, visual appearance and weight loss on heating the glassware under test conditions may be used. Glassware needs to show no signs of discoloration or particulates on the surface. The weight of glassware must not change by more than  $\pm 0.5$  mg between cleaning and heating/cooling cycles. Detergent cleaning avoids the potential hazards and inconveniences related to handling corrosive chromic acid solutions; this procedure remains the reference cleaning practice and as such may function as an alternate to the preferred procedure, cleaning with detergent solutions.

9.2 Remove container and cover from the cleaning solution by means of corrosion-resistant steel forceps and handle only with forceps thereafter. Wash thoroughly, first with tap water, then with distilled water, and dry in an oven at 100 to 150°C for at least 1 h.

9.3 Drain any gasoline from the pressure vessel and wipe the inside of the pressure vessel and pressure vessel closure, first with a clean cloth moistened with gum solvent and then with a clean dry cloth. Remove the filler rod from the stem and carefully clean any gum or gasoline from the stem, rod, and needle valve with gum solvent. The pressure vessel, the valve, and all connecting lines shall be thoroughly dry before each test is started. (**Warning**—Volatile peroxides which may have formed during a previous test may accumulate in the equipment, producing a potentially explosive environment. Special care in cleaning after each test is needed to ensure the filler rod, stem, and needle valve are free of these peroxides.)

## 10. Procedure

10.1 Bring the pressure vessel and the gasoline to be tested to a temperature of 15 to 25°C. Place the glass sample container in the pressure vessel and add  $50 \pm 1$  mL of test specimen. Alternatively, transfer  $50 \pm 1$  mL of test specimen into the glass sample container first, before placing the glass sample container into the pressure vessel. Cover the sample container, close the pressure vessel, and using a quick release air coupling introduce oxygen until a pressure of 690 to 705 kPa is attained. Allow the gas in the bomb to escape slowly in order to flush out the air originally present (*Release the pressure at a slow uniform rate through the needle valve at a rate not to exceed 345 kPa per min*). Introduce oxygen again until a pressure of 690 to 705 kPa is attained and observe for leaks, ignoring an initial rapid drop in pressure (generally not over 40 kPa) which may be observed because of the solution of oxygen in the sample. When the rate of pressure drop does not exceed 7 kPa in 10 min, assume the absence of leaks and proceed with the test without repressuring.

10.2 Place the charged pressure vessel in the vigorously boiling water bath, or appropriate liquid bath equipped with mechanical stirring, being careful to avoid shaking, and record the time of immersion as the starting time. Maintain the temperature of the liquid bath between 98 and 102°C. Observe the temperature to the nearest 0.1°C at intervals during the test, and record the average temperature to the nearest 0.1°C as the temperature of the test. Make a continuous record of the pressures in the pressure vessel, or if an indicating pressure gage is used, take pressure readings at 15-min intervals or less. If, during the initial 30 min of the test, a leak develops (as indicated by a steady drop in pressure considerably in excess of 14 kPa in 15 min) discard the test. Continue the test until reaching a point preceded by a pressure drop of exactly 14 kPa in 15 min and succeeded by a drop of not less than 14 kPa in 15 min or until the induction period exceeds the product specification. If no breakpoint is observed at the termination of the test, refer to Section **12** for reporting the result. (**Warning**—If a boiling water bath is used and the test is made in a region where the atmospheric pressure is consistently below normal (101.3 kPa), it is permissible to add a liquid with

higher boiling point, such as ethylene glycol, to the water in order to maintain the operating temperature of the bath in the neighborhood of 100°C. If a liquid other than water is used, be sure the liquid is compatible with the seals in the pressure vessel.)

10.3 Record the number of minutes from the time the pressure vessel is placed in the bath until the breakpoint has been reached as the observed induction period at the temperature of the test.

10.4 Cool the pressure vessel to approximately room temperature within 30 min after removal from the bath, using ambient air or water  $\leq 35^\circ\text{C}$ , before releasing the pressure slowly from the pressure vessel through the needle valve at a rate not exceeding 345 kPa per minute. Wash the pressure vessel and sample container in preparation for the next test.

## 11. Calculation

11.1 The number of minutes from the time the pressure vessel is placed in the bath until the breakpoint has been reached is the observed induction period at the temperature of the test.

11.2 *Method of Calculation*—Calculate the induction period at 100°C from one of the following equations: When the test temperature is above 100°C:

$$\text{Induction period at } 100^\circ\text{C} = (IP_t)(1 + 0.101(t_a - 100)) \quad (1)$$

When the test temperature is below 100°C:

$$\text{Induction period at } 100^\circ\text{C, min} = (IP_t)/(1 + 0.101(100 - t_b)) \quad (2)$$

where:

$IP_t$  = induction period, min, at the temperature of the test,

$t_a$  = test temperature when above 100°C, °C, and

$t_b$  = test temperature when below 100°C, °C.

## 12. Report

12.1 Report the Induction period at 100°C, calculated as in 11.2 to the nearest 1 min.

12.2 If the test was stopped prior to observing the pressure drop required in 10.2, but after the product specification was exceeded, then report the result as being greater than N min, where N is the product specification in min.

12.3 If a slow oxidation of the sample is occurring rather than a breakpoint as defined in 10.2, then report the sample as a slow oxidizing fuel with the total time for the test and the overall pressure drop from the start of the test. A precision and bias statement has not been determined for this case.

## 13. Precision and Bias

13.1 The precision of the test method as determined by statistical examination of interlaboratory test results is as follows:

13.1.1 *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: 5 %.

13.1.2 *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: 10 %.

13.2 *Bias*—There being no criteria for measuring bias in these test-product combinations, no statement of bias can be made.

NOTE 4—The precision values given above for induction period were obtained using a boiling water bath only as the heat source. Therefore, these precision values are not necessarily applicable to induction period results obtained using other heat sources.

## 14. Keywords

14.1 breakpoint; gasoline; induction period; oxidation stability

## ANNEX

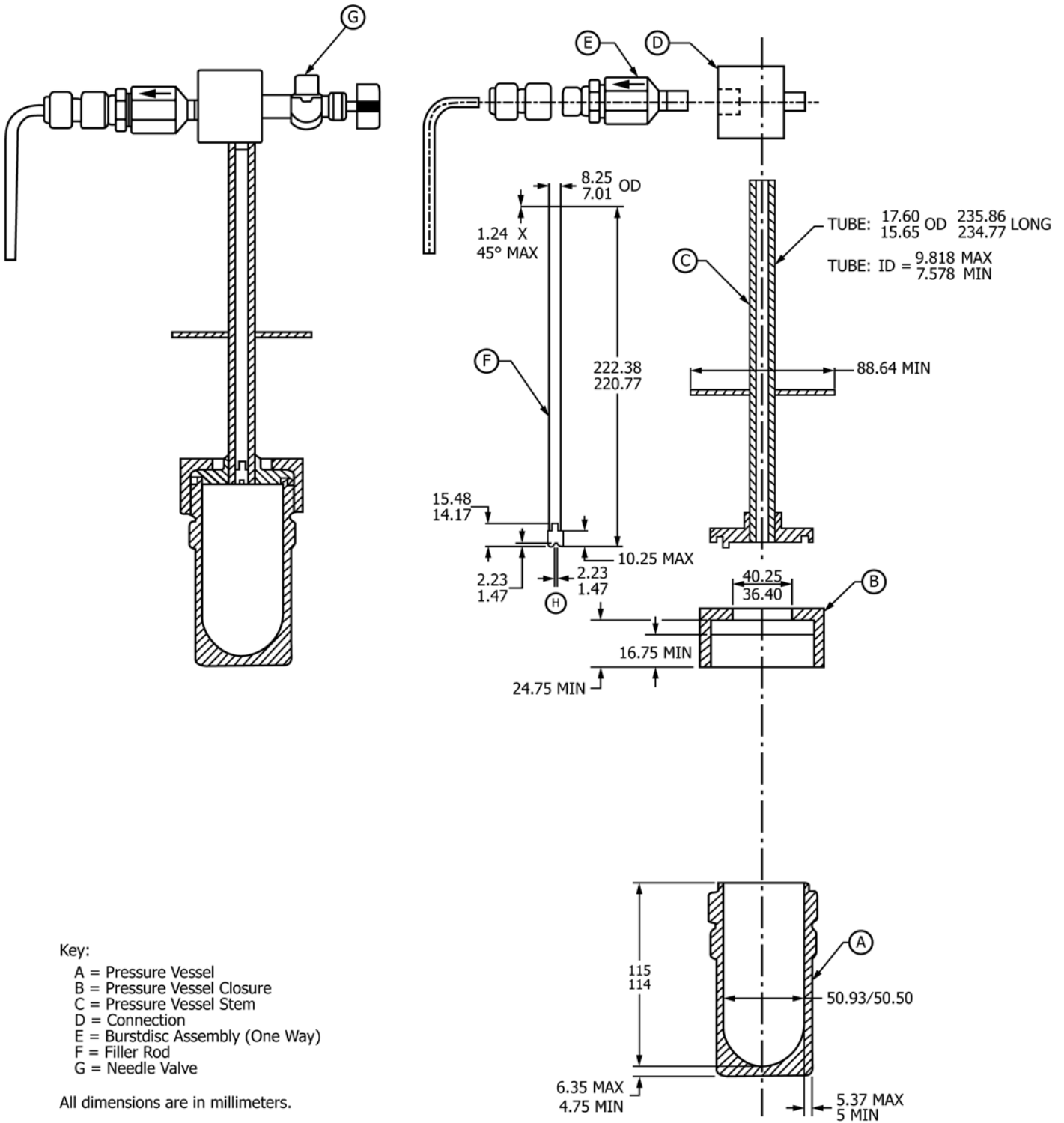
### (Mandatory Information)

#### A1. APPARATUS

##### A1.1 Apparatus

A1.1.1 *Pressure Vessel*—The pressure vessel shall be made of corrosion-resistant steel with the inside dimensions of the portion that encloses the reacting gasoline-oxygen mixture conforming substantially to those shown in Fig. A1.1. Fig. A1.1 is a composite drawing of specific pressure vessels and related apparatus for performing Test Method D525 as made by various manufacturers. As such, ranges of specific dimensions have been given and are not intended to be precise. Pressure

vessels conforming to Test Method D525/1980-1995 as well as IP 40 are also suitable, but the specified burst-disc shall be attached. Small variations in external dimensions are not considered to significantly impact the results of the test, but specific studies to document potential effects, if any, have not been carried out. A minimum wall thickness of 5 mm in the pressure vessel is specified for safety reasons. (**Warning**—Pressure vessel assembly components obtained from different suppliers/manufacturers may not be compatible.)



NOTE 1—The dimensions on the burst disc outlet pipe are: internal diameter – 3.7 mm id, min radius – 14.95 mm, min length – 35 by 75 mm, typical or approximate.

FIG. A1.1 Composite Pressure Vessel for Oxidation Stability Test of Gasoline

A1.1.1.1 The interior surfaces of the pressure vessel and pressure vessel closure shall have a high polish such as a surface roughness of 0.20 to 0.40 micrometres, to facilitate cleaning and to prevent corrosion.

A1.1.1.2 Other structural details, such as method of closure, (polygonal or knurled) gasket material, and outside dimensions are optional provided the limitations listed in A1.1.1.3 and A1.1.1.4 are observed:

NOTE A1.1—Initial testing and periodic examination of the pressure vessel should be carried out to ensure its fitness for service.

A1.1.1.3 The pressure vessel shall be constructed to withstand a working pressure of 1240 kPa at 100°C, with an ultimate strength at least equal to that of a pressure vessel constructed of 18 % (m/m) chromium, 8 % (m/m) nickel-alloy steel. A suitable material is an alloy steel conforming to the specification for 303 or 304 stainless steels.

A1.1.1.4 The closure shall be capable of making a seal that will not leak when the pressure vessel is filled with oxygen to 690 to 705 kPa at 15 to 25°C and plunged into a bath at 100°C. It is preferable that the closure ring be constructed from an alloy different from that of the body if the mating threads of the two parts are to move with respect to each other when the tightening load is applied.

A1.1.2 *Gasket*—Any suitable gasket material will pass the test described in A1.1.2.1.

A1.1.2.1 Place a gasket of the type under test in the pressure vessel in the absence of gasoline and use a similar gasket to make the seal with the pressure vessel closure. Fill the pressure vessel with oxygen at a pressure of 690 to 705 kPa and immerse in a bath at about 100°C. If the pressure does not drop more than 14 kPa from the maximum in a 24-h period with the bath temperature constant at  $\pm 1.0^\circ\text{C}$ , the gasket shall be considered satisfactory.

A1.1.3 *Glass Sample Container and Cover*, conforming to the dimensions given in Fig. A1.2.

A1.1.3.1 *Cover*, capable of keeping material refluxing back through the pressure vessel stem from entering the sample but not to prevent free access of oxygen to the sample. One such example of a cover is shown in Fig. A1.2, where the identified dimensions have been found suitable to use.

A1.1.4 *Pressure Vessel Stem*—A stem with a filler rod, constructed of the same material as the pressure vessel closure, and having dimensions conforming to those shown in Fig. A1.1.

A1.1.4.1 The filler rod and the inside of the stem shall have a high polish such as surface roughness of 0.20 to 0.40 micrometres to facilitate cleaning and prevent corrosion. The stem shall be fitted, in the position shown in Fig. A1.1, with a circular metal plate 89 mm in diameter to serve as a closure for the bath when the pressure vessel is in place.

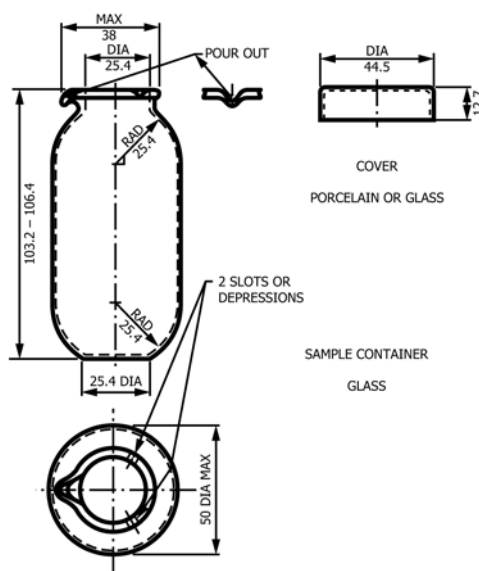
A1.1.5 *Burst-disc Assembly*—The stem shall be fitted with a stainless steel burst-disc assembly which will rupture if subjected to a pressure greater than 1530 kPa  $\pm 10\%$ . The assembly shall be mechanically designed to ensure that it cannot be incorrectly fitted. (**Warning**—Provision shall be made to safely vent any expelled gases or flames away from the operator, other personnel, or flammable materials as a safety precaution if the burst disc ruptures.)

A1.1.6 *Connection*—Provision shall be made for connecting a pressure gage and a tightly closing needle valve to the pressure vessel stem as shown in Fig. A1.1. A quick release air coupling fitted to the needle valve shall be used to facilitate oxygen entry to the pressure vessel.

A1.1.7 *Needle Valve*—A control valve suitable for complete shut off but fitted with a finely tapered needle and orifice.

NOTE A1.2—The valve should be used while purging, pressurizing, and exhausting the pressure vessel with oxygen.

A1.1.8 *Pressure Gage*—An indicating or a recording type gage reading to at least 1380 kPa. Pressure transducers and digital readout devices may be used provided that accuracy of measurement is not adversely affected.



NOTE 1—All dimensions are millimetres.

NOTE 2—One of the V-shaped depressions shall be depressed sufficiently to serve as a pouring lip.

NOTE 3—Glass sample container, the dimensions on the flat bottom, inner diameter (near the opening), and the internal radius of the curved sides are  $25.4 \pm 2.0$  mm. The width of the container at its widest point is  $50 \pm 2$  mm.

NOTE 4—Glass sample cover, the outside diameter is  $44.5 \pm 2.0$  mm and the height is 12.7 mm, max.

FIG. A1.2 Glass Sample Container and Cover (Glass or Porcelain)

A1.1.8.1 Any half of the scale interval (that is, 345 kPa) between 690 and 1380 kPa shall be at least 25 mm in length measured along the arc of the scale. The intervals of division shall be 35 kPa or less. The accuracy shall be 1 % or less of the total scale interval. Other suitable equivalent metric gages may be used.

A1.1.8.2 The gage shall be connected to the pressure vessel directly or by flexible metal or metal sheathed gas-resistant polymeric tubing having a pressure rating to satisfy the above conditions. The total volume of the flexible tubing, connections, and stem with filler rod in place, shall not exceed 30 mL. (**Warning**—When ordering equipment for this test, the manufacturer should be requested to ensure pressure gage and needle valve are suitable for use with oxygen.)

A1.1.9 *Oxidation Bath*—The liquid bath shall have a capacity of not less than 18 L for one pressure vessel, plus 8 L for each additional pressure vessel in multiple assemblies, and be of such dimensions that the maintained depth of the bath liquid is not less than 290 mm. A non-self-resettable device shall be fitted on all baths supplied to ensure that the heater is switched off if the bath liquid falls below a safe level and to prevent the bath from boiling dry. Users of older baths without this device are strongly urged to have the equipment retrofitted to ensure safe operation.

A1.1.9.1 The top of the bath shall have openings of a suitable diameter to accommodate the pressure vessel and to fit the cover plate fastened to the pressure vessel stem. If a

thermometer is being used to monitor bath temperature, a thermometer well shall be provided in such a position that the 97°C mark of the thermometer is above the cover of the bath. For other temperature sensing devices, the bath shall have provisions to allow for the temperature to be monitored appropriately.

A1.1.9.2 When in place, the top of the pressure vessel closure shall be submerged at least 50 mm below the surface of the bath liquid.

A1.1.9.3 Auxiliary lids are needed to cover the openings when the pressure vessels are not in the baths. The bath shall be provided with a condenser and source of heat to maintain the bath water boiling vigorously. If a liquid medium other than water is used, an appropriate mechanical stirrer/mixer shall be used to maintain uniformity of the liquid bath at  $100 \pm 2^\circ\text{C}$ .

NOTE A1.3—Electric heating blocks are known to be used. These blocks can have heat capacities, heating rates, and heat transfer characteristics which differ from those of a liquid bath. An electric heating block may be used in place of a liquid bath as long as the sample heating rate and sample temperature are demonstrated to be equivalent to that of the liquid bath.

A1.1.10 *Thermometer*, having a range from 95 to 103°C, conforming to the requirements prescribed in Specification E1 or in the specifications for IP thermometers. Other temperature sensing devices that cover the temperature range of interest, such as thermocouples or platinum resistance thermometers, that can provide equivalent or better accuracy and precision, may be used in place of the thermometers specified in 6.2.

## APPENDIX

### (Nonmandatory Information)

#### X1. TEMPERATURE CALIBRATION OF DRY BLOCK BATH

##### X1.1 General

X1.1.1 *Principle*—The sample temperature inside the pressure vessel (during a simulated test, but without any pressure applied) is measured and compared to the temperature of the dry block bath after an equilibrium period of one hour. If a temperature difference is measured, the temperature of the dry block bath is adjusted so that the sample temperature equilibrates at the correct temperature.

##### X1.2 Apparatus—Temperature Calibration Set

X1.2.1 *Temperature Probe*—Calibrated, consisting of: Mantle-Thermocouple (for example, type “K” (NiCr-Ni), according to IEC 584, class 1), minimum of 400 mm in length with a diameter of 3 mm and a tapered tip with a minimum length of 18 mm and a 1.5 mm diameter (see Fig. X1.1).

X1.2.2 *Temperature Probe Adapter*—Adapter to clamp the thermocouple at the correct position and height in the vessel.

X1.2.3 *Temperature Readout*—Measuring instrument or data recorder for displaying or storing the sample temperature.

X1.2.4 *Centering Cover*—PTFE cover with 3 mm hole for centering the thermocouple inside the sample container.

##### X1.3 Procedure

X1.3.1 Remove the filler rod from the pressure vessel stem.

X1.3.2 Screw the temperature probe adapter into the female thread on top of pressure vessel (where the pressure detector is normally fitted).

X1.3.3 Fill the sample container with 50 mL of a low-viscosity oil sample.

X1.3.4 Replace the original glass cover of the sample container with the centering cover.

X1.3.5 Insert the thermocouple through the pressure vessel stem and clamp it with the probe adapter so that the tip is approximately 15 mm above the bottom of the sample container and centered in the sample.

X1.3.6 Insert the pressure vessel into the preheated Dry Block Bath at the specified and stable test temperature.

X1.3.7 Allow the device to stabilize for one hour and note or record the temperature inside the sample after this period is finished.

X1.3.8 If an offset is found between the temperature of the sample and the temperature of the dry block bath, adjust the

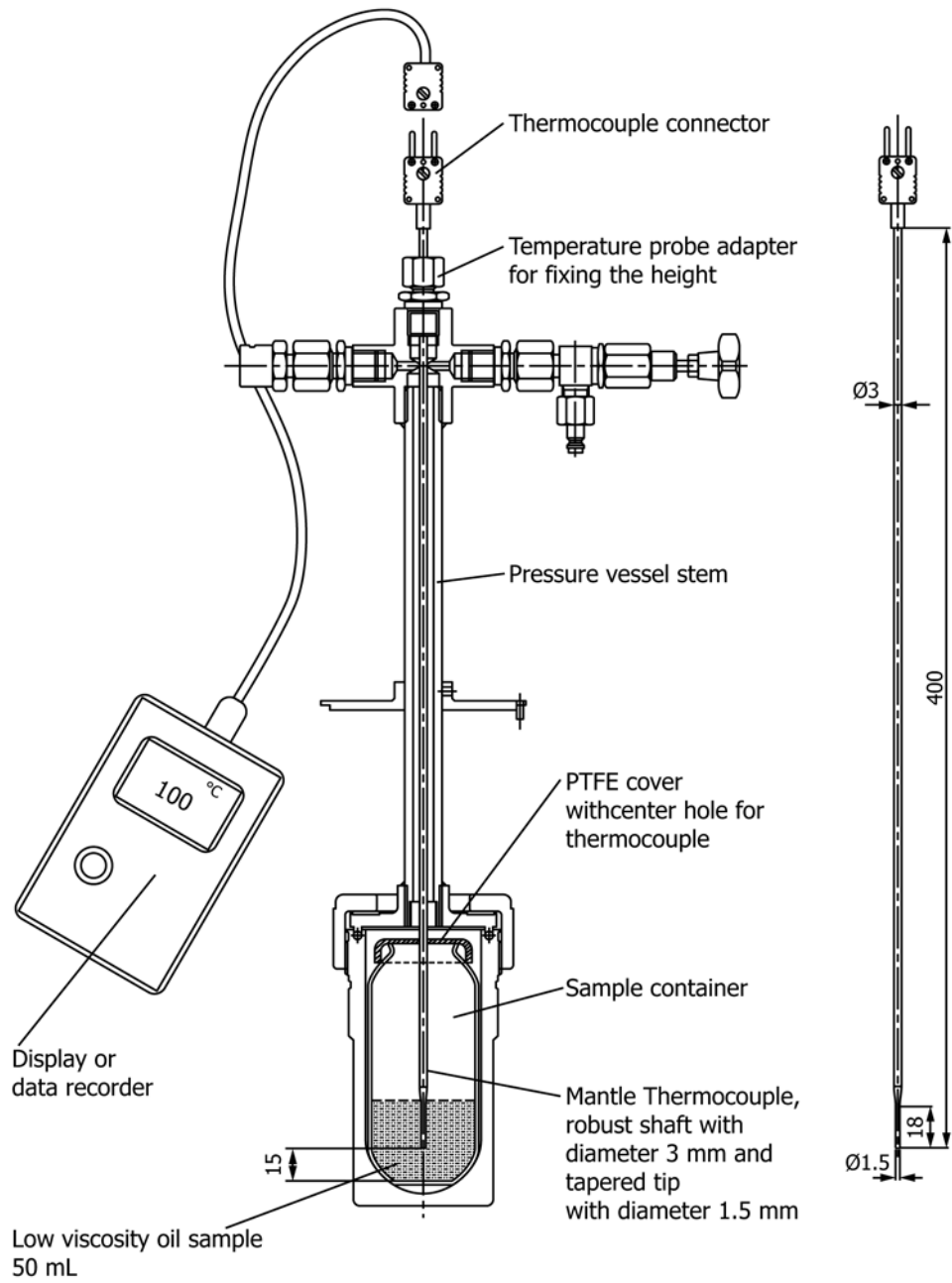


FIG. X1.1 Temperature Calibration Set in Pressure Vessel

temperature of the dry block bath so that the sample will reach the correct test temperature.

X1.3.9 Repeat the steps of X1.3, Procedure, to verify that the sample equilibrates at the correct test temperature.

NOTE X1.1—Charging with oxygen is neither necessary nor possible with this calibration procedure.

**SUMMARY OF CHANGES**

Subcommittee D02.14 has identified the location of selected changes to this standard since the last issue (D525–12) that may impact the use of this standard.

(1) Added **Appendix X1**.

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