Standard Test Method for
Oxidation Stability of Spark Ignition Fuel—Rapid Small Scale Oxidation Test (RSSOT)[1]

This standard is issued under the fixed designation D7525; 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 laboratory test method covers the quantitative determination of the stability of spark ignition fuel, including those containing alcohols or other oxygenates, under accelerated oxidation conditions, by an automatic instrument (**Warning—**This test method is not intended for determining the stability of gasoline components, particularly those with a high percentage of low boiling unsaturated compounds, as these can cause explosive conditions with the apparatus.[2])

1.2 This test method measures the induction period, under specified conditions, which can be used as an indication of the oxidation and storage stability of spark ignition fuel.

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

1.4 *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:*[3]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *break point, n*—pressure in the test apparatus, which is 10 % below the maximum pressure of the actual test run.

3.1.2 *induction period, n*—time elapsed between starting the heating procedure of the sample vessel and the break point, measured in minutes.

4. Summary of Test Method

4.1 A 5 mL sample is introduced into a pressure vessel which is then charged with oxygen to 500 kPa at a temperature of 15 to 25°C. The test is initiated by starting the heater and heating the pressure vessel to a temperature of 140°C.

4.2 The pressure is recorded continuously until the break-point is reached. Alternatively, the test may be terminated when a predetermined minimum requirement is reached.

5. Significance and Use

5.1 The induction period may be used as an indication of the oxidation and storage stability of spark ignition fuel.

5.2 Compared to some other oxidation and storage stability test methods, this test method uses a small sample and gives a result in a short time period.

6. Apparatus

6.1 *General*—This test method uses an automatically controlled oxidation tester[4] (Fig. 1) comprising an oxidation pressure vessel containing a test sample cup capable of being rapidly heated, fitted with a pressure sensor capable of measuring pressures up to 2000 kPa and a temperature sensor capable of reading to 0.1°C. Pressure and temperature in the oxidation vessel are recorded continuously during the test. The oxidation pressure vessel is fitted with filling and relief valves and a means of automatically releasing the pressure at the end of the test. The integrated cooling fan cools the pressure vessel

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[1] 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.

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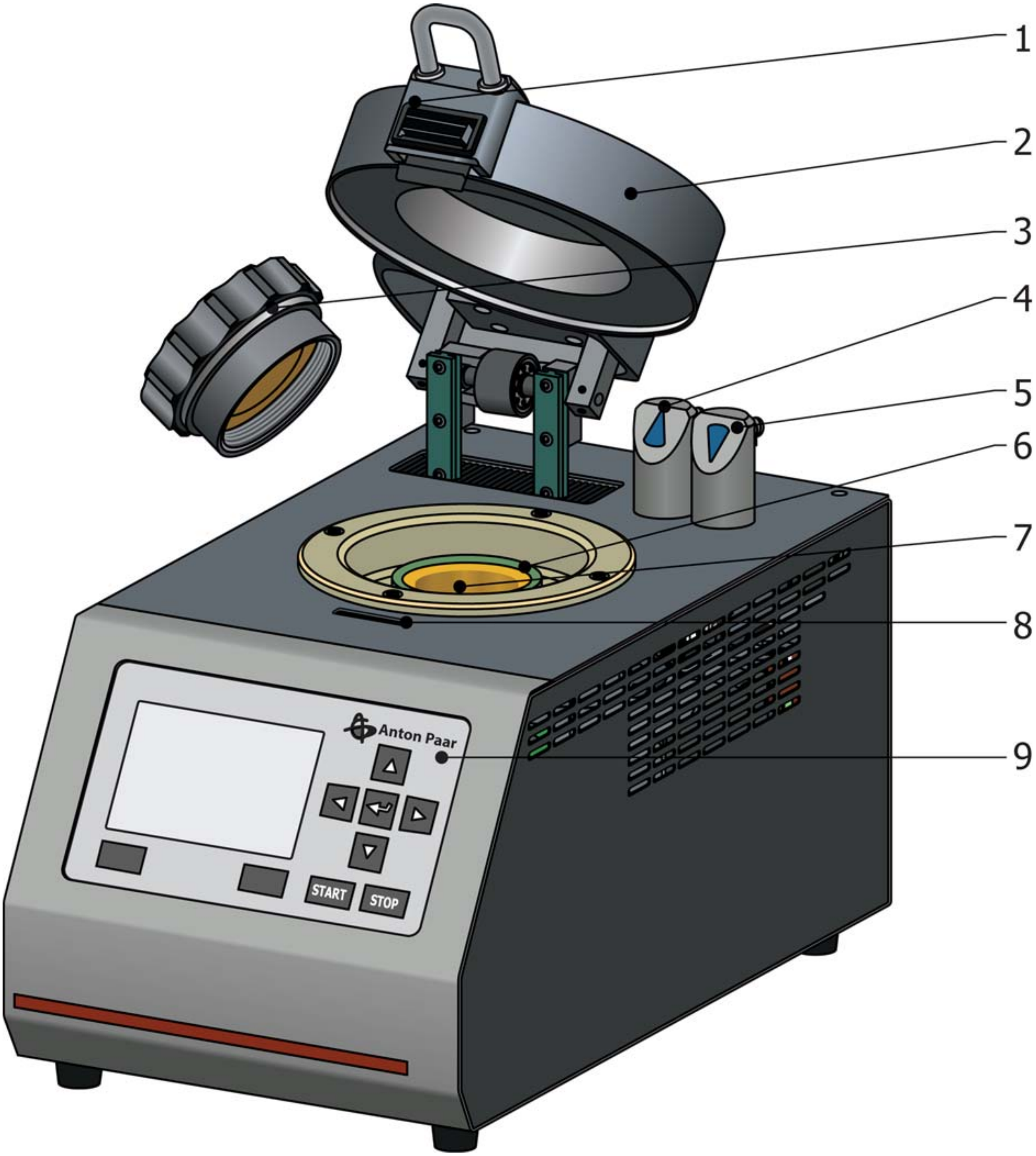
[2] Further information can be found in the June 1978, January 1979, and June 1986 editions of the *Institute of Petroleum Review*.

[3] 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.

[4] The sole source of supply of the apparatus known to the committee at this time is Anton Paar's PetroOxy apparatus, available from Anton Paar ProveTec GmbH, Ludwig-Erhard-Ring 13, 15827 Blankenfelde-Mahlow, Germany, www.anton-paar.com. 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.

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



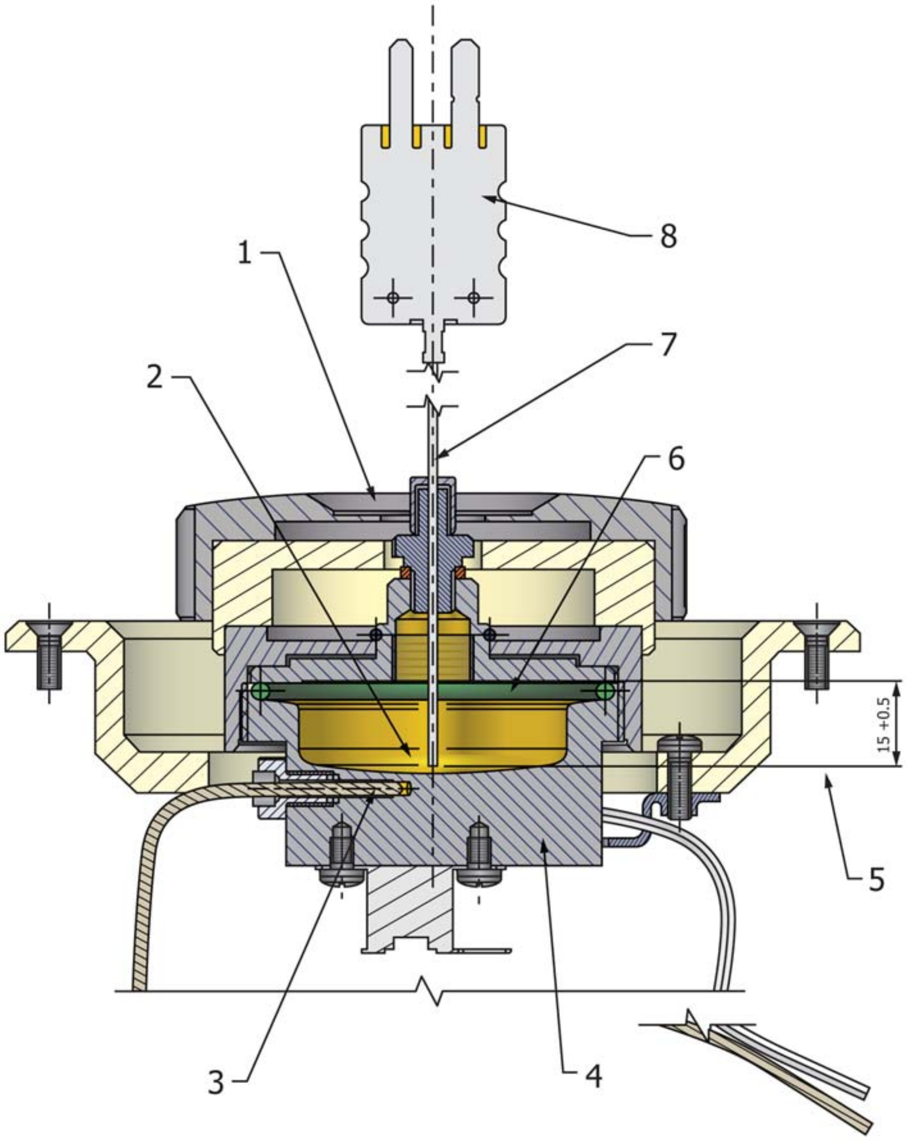
1. Unlocking mechanism for insulating hood
2. Safety and insulating hood
3. Screw cap for closing the test container
4. Oxygen outlet
5. Oxygen inlet
6. “O-ring” seal for test sample cup
7. Test sample cup
8. Locking mechanism for insulating hood
9. Operating panel with display

FIG. 1 Schematic of Apparatus for Rapid Small Scale Oxidation Test

from the test temperature to ambient temperature by a flow of air. See Annex A1 for detailed information.

6.2 *Volumetric Device,* clean and free from contaminations of previous samples, capable of delivering 5.0 ± 0.1 mL.

6.3 *Temperature Calibration Equipment,* comprising a cover and a temperature calibration sensor (Fig. 2). The temperature calibration sensor is fixed to a depth of 15 ± 0.5 mm. The temperature calibration sensor, calibrated to the



1. Calibration cover with duct for temperature calibration sensor
2. Calibration fluid
3. Temperature sensor for block temperature
4. Heating block
5. Immersion depth of temperature calibration sensor and bottom of calibration cover
6. Seal
7. Temperature calibration sensor
8. Connector plug to measuring device

FIG. 2 Temperature Calibration Equipment

nearest 0.1°C, is calibrated by an approved calibration service, such as one that is traceable to the National Institute of Standards and Technology (NIST) or to a national authority in the country in which the equipment is used or manufactured.

6.4 *Pressure Calibration Equipment,* comprising a pressure calibration sensor (Fig. 3). The pressure calibration senor is calibrated to the nearest 10 kPa, by an approved calibration service, such as one that is traceable to the National Institute of Standards and Technology (NIST) or to a national authority in the country in which the equipment is used or manufactured.

7. Reagents and Materials

7.1 Solvent for the removal of oxidation residues from the test vessel. The solvent shall be of suitable purity to leave no residue on the apparatus. Ethanol of 94 % minimum purity has been found to be suitable. Other solvents, such as a mixture of

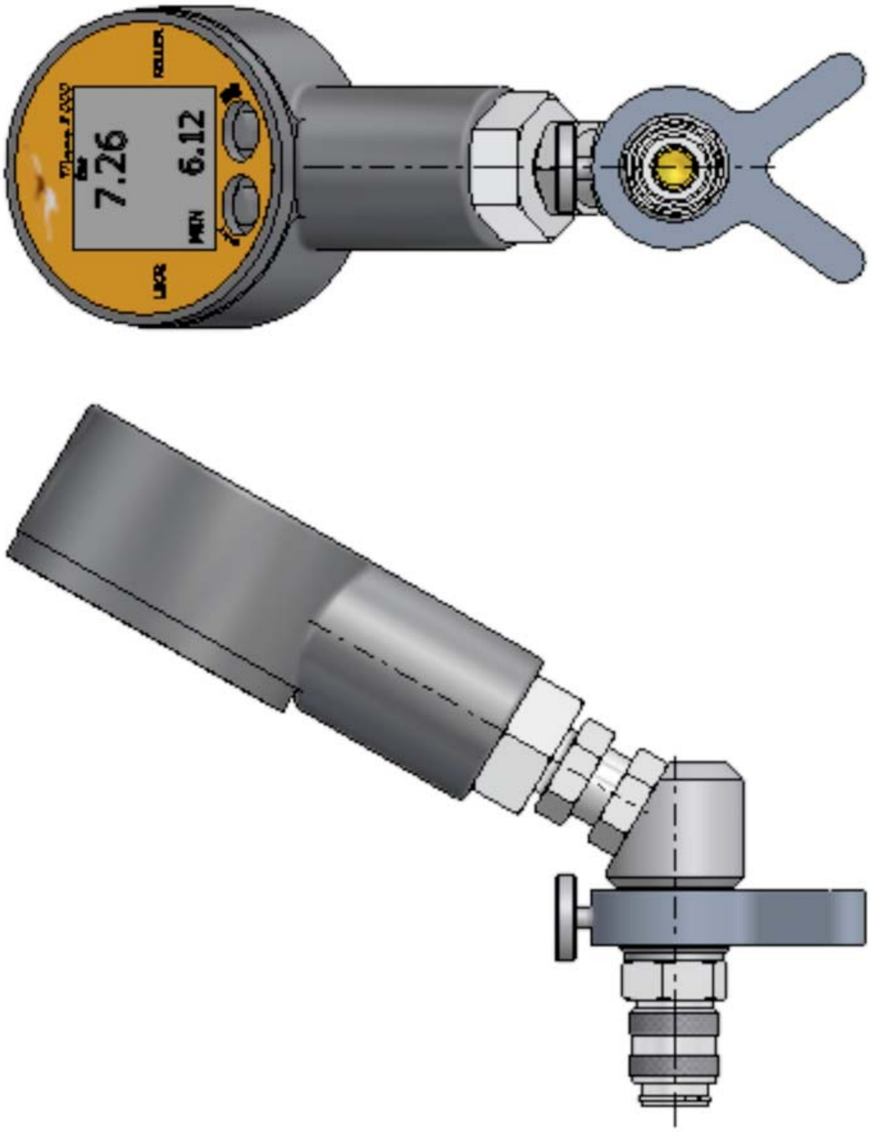


FIG. 3 Pressure Calibration Equipment

equal volumes of toluene and acetone, may be used if shown to meet the requirements for the removal of oxidation residues from the test vessel without leaving any residue in the sample test cup.

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

7.3 *Lint-Free Cleaning Tissue*—For sensitive surfaces; that will not scratch the surface.

7.4 *"O-ring" Seal*—See A1.2.

7.5 *Temperature Calibration Fluid*—Stable middle distillate fuel liquid with flash point above +60°C and boiling-point above +150°C.

8. Hazards

8.1 (**Warning—**To provide protection against possible explosive rupture of the pressure vessel and hazards relating to hot and flammable fuels, the apparatus shall be operated behind an appropriate safety shield.)

9. Sampling

9.1 Sample in accordance with Practices D4057 or D4177.

10. Preparation of Apparatus

10.1 Remove the previous sample by means of a pipette or similar device.

10.2 Remove the used "O-ring" seal and discard.

NOTE 1—To avoid contamination of the new test, it is necessary to discard the used "O-ring" seal, because it might be soaked with oxidation products from the previous test.

10.3 Wipe the test sample cup, the seal groove and the cover of the test vessel with lint-free cleaning tissue (7.3) soaked with solvent until free of gum or other oxidation residues.

10.4 Allow the test sample cup and cover to dry in air and visually inspect for cleanliness.

NOTE 2—Compressed air is generally unsuitable to speed up evaporation of solvent because it can contain traces of oil that could contaminate the next test.

10.5 Insert a new "O-ring" seal.

11. Calibration

11.1 Recalibrate the tester every 12 months for correct temperature and pressure detection.

11.2 Calibration of temperature indicator.

11.2.1 Calibrate the temperature sensor (A1.6) to the nearest 0.1°C using the temperature calibration equipment (6.3) according to A2.1.

11.3 Calibration of pressure detector.

11.3.1 Calibrate the pressure sensor (A1.5) to the nearest 10 kPa, using the pressure calibration equipment (6.4) according to A2.2.

11.4 Verify, at least every 3 months, that the heater is operating properly and the pressure vessel is reaching 140 ± 0.5°C within 5 min.

12. Procedure

12.1 Switch the apparatus on. Bring the pressure vessel and the spark ignition fuel sample to be tested to a temperature of 15 to 25°C.

12.2 Using a volumetric device (6.2), place 5 ± 0.1 mL of the sample into the test sample cup (Fig. 1, No. 7).

12.3 Cover the test sample cup with the screw-cap (Fig. 1, No. 3), and close the pressure vessel.

12.4 Introduce oxygen (7.2) into the pressure vessel until a pressure of 500 ± 5 kPa is attained and stabilized over 20 s.

12.5 Start the heater with no delay between charging with oxygen and starting the test. The apparatus automatically starts the timer.

12.6 The apparatus brings the oxidation pressure vessel and test specimen to 140 ± 0.5°C within 5 min.

12.7 If, during the initial 5 min of the test, a steady drop in pressure is observed, discontinue the test and discard the test specimen.

12.7.1 Under no circumstances may the leakage rate exceed a value of 2 kPa/h. If the leakage rate shows an increase, check the following components:

O-ring for damage or residues of samples,
Surface of the sample cup for damage, and
Sample cup for sample residues.

12.7.2 Contact the manufacturer to resolve leakage problems from other parts of the instrument.

12.8 The apparatus automatically records the temperature (to the nearest 0.1°C) and pressure (to the nearest 1 kPa) of the oxidation vessel continuously.

12.9 The test apparatus will automatically terminate the test when the pressure readings show a 10% drop from the maximum observed pressure. This is the breakpoint.

12.10 Record the induction period to the nearest 1 min.

12.11 Alternatively, terminate the test when the test time exceeds a predetermined minimum requirement.

NOTE 3—The apparatus will automatically switch on the fan to cool the pressure vessel to approximately room temperature. When the apparatus has cooled sufficiently, the apparatus automatically releases the pressure slowly from the pressure vessel through the valve at a rate not exceeding 345 kPa/min.

12.12 When the pressure release process has finished, open the apparatus and clean according to Section 10.

13. Report

13.1 Report the induction period to the nearest whole minute, and reference this test method. If the test was stopped (12.11) prior to observing the pressure drop required in 12.9, report induction period (referencing this test method) as greater than N minutes, where N is the predetermined minimum time within the induction period.

14. Precision and Bias

14.1 The precision statement was determined in an interlaboratory study (#0542) in 2011 with seven participating laboratories and eleven samples.

14.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of this test method, exceed the following only in one case in twenty.

$$r = 0.3903 \cdot X^{0.6} \tag{1}$$

where:

X = the mean of the two test results expressed in minutes, rounded to the nearest 0.01 min.

NOTE 4—The degrees of freedom associated with the repeatability estimate from this round robin study are 29. Since the minimum requirement of 30 (in accordance with Practice D6300) is not met, users are cautioned that the actual repeatability may be significantly different than these estimates.

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

$$R = 1.0212 \cdot X^{0.6} \tag{2}$$

where:

X = the mean of the two test results expressed in minutes, rounded to the nearest 0.01 min.

NOTE 5—Supporting data have been filed at ASTM International

Headquarters and may be obtained by requesting Research Report RR:D02-1783.[5]

14.2 *Bias*—Since the value of oxidation stability measured by this test method is defined solely by this test method, no statement of bias can be made.

15. Keywords

15.1 breakpoint; gasoline; induction period; oxidation stability; oxygen uptake; spark ignition fuel

ANNEXES

(Mandatory Information)

A1. AUTOMATICALLY CONTROLLED OXIDATION TESTER

A1.1 Pressure Vessel and Closure

A1.1.1 Internal volume without sample: 20 mL. Inner diameter: 47 ± 0.15 mm.

A1.1.2 Made of corrosion-resistant material and constructed to withstand a working pressure of 1800 kPa.

A1.1.3 Interior surfaces: smooth (polished), chemically inert surface to facilitate cleaning and prevent corrosion.

A1.2 "O-ring"

A1.2.1 *Seal for Test Sample Cup*—Made of material being resistant to oxygen, spark ignition fuel components and heat, typically fluoro-elastomer FPM/FKM (commonly known as Viton[6]), coated with PTFE.

A1.3 Heating

A1.3.1 Installed power rating: 500 W. Typically electric heating, near the bottom of the pressure vessel, to facilitate best heat transfer to the sample.

A1.3.2 The heater control shall be capable of maintaining the test cup temperature within ±0.5°C at the test temperatures of 140°C.

A1.4 Valves

A1.4.1 Solenoid valves with small dead volume and orifice sizes and short cycling rates.

A1.5 Pressure Sensor

A1.5.1 Absolute or relative pressure transducer.

A1.5.2 *Pressure Range*—0 to 2000 kPa (minimum).

A1.5.3 *Accuracy*—±3 % full-scale span.

A1.5.4 *Typical Sensitivity*—1 to 25 mV/kPa.

A1.6 Temperature Sensor

A1.6.1 *Platinum Resistance Thermometer*—accuracy ± (0.1 + 0.0017 x (t)), where (t) is the actual temperature in °C, range up to 200°C.

A1.7 Connecting Pipes

A1.7.1 Small orifices, typical inner diameter: 0.5 to 1.0 mm.

A1.8 Cooling Fan

A1.8.1 Capable of cooling the pressure vessel from the test temperature to ambient temperature by applying a stream of air to the outside of the pressure vessel.

A1.9 General Requirements

A1.9.1 All equipment in contact with the sample and oxygen shall be made of corrosion-resistant materials.

A1.9.2 The pressure vessel shall have an over-temperature safeguard.

A1.9.3 A cooling device for rapid cooling is recommended.

A1.9.4 A safety and isolation hood above the pressure vessel closure is recommended (see Fig. 1).

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1783. Contact ASTM Customer Service at service@astm.org.

[6] Registered trademark of DuPont Performance Elastomers, 300 Bellevue Parkway, Suite 300, Wilmington, DE 19809.

A2. INSTRUMENT CALIBRATION

A2.1 Temperature Calibration

A2.1.1 Calibrate temperature sensor (A1.6) at ambient and at 140°C.

A2.1.2 Fill the cup with 10 ± 0.1 mL of the calibration fluid (7.5).

A2.1.3 Ensure that the temperature calibration sensor's depth is fixed at 15 ± 0.5 mm (Fig. 2, No. 5).

A2.1.4 Cover the test sample cup with the temperature calibration equipment (6.3) (Fig. 2).

A2.1.5 When the temperature is stabilized, record the temperatures indicated by the temperature sensor (A1.6) and the temperature calibration sensor.

A2.1.6 Calculate offset and gain according to manufacturer's manual and change, if required, according to manufacturer's manual.

A2.2 Pressure Calibration

A2.2.1 Calibrate the pressure sensor (A1.5) at two pressures.

A2.2.2 Cover the empty sample cup with the screw-cap (Fig. 1, No. 3).

A2.2.3 Connect the oxygen filling line to the instrument, and pressurize the test cell to 700 kPa.

A2.2.4 Close the filling valve, remove the oxygen filling line, and replace it by the calibration equipment (Fig. 3)(6.4).

A2.2.5 Start the calibration procedure in accordance with the manufacturer's manual.

A2.2.6 Open the valve to the pressure calibration sensor. Pressure will slightly drop.

A2.2.7 Record the pressures indicated by the pressure sensor (A1.5) and the pressure calibration sensor.

A2.2.8 Close the valve to the pressure calibration equipment (6.4).

A2.2.9 Reduce the pressure in the test cell to approximately 20 kPa and open the valve to the calibration equipment. Pressure will slightly increase to 90 to 100 kPa.

A2.2.10 Record the pressures indicated by the pressure sensor (A1.5) and the pressure calibration sensor.

A2.2.11 Release the pressure.

A2.2.12 Calculate offset and gain in accordance with the manufacturer's manual, and change if required by the manufacturer's manual.

SUMMARY OF CHANGES

Subcommittee D02.14 has identified the location of selected changes to this standard since the last issue (D7525 –) that may impact the use of this standard. (Approved Oct. 1, 2014.)

(1) Revised Section 14, with new 14.1, new 14.1.1, new Note 4, new 14.1.2, and new Note 5.