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Standard Test Method for Pressure Calibration of Thermal Analyzers[1]

This standard is issued under the fixed designation E2744; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes the calibration or performance confirmation of the electronic pressure signals from thermal analysis apparatus.

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

1.3 There is no ISO standard equivalent to this test method.

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:*[2]

D5483 Test Method for Oxidation Induction Time of Lubricating Greases by Pressure Differential Scanning Calorimetry
D6186 Test Method for Oxidation Induction Time of Lubricating Oils by Pressure Differential Scanning Calorimetry (PDSC)
D5720 Practice for Static Calibration of Electronic Transducer-Based Pressure Measurement Systems for Geotechnical Purposes
D5885 Test Method for Oxidative Induction Time of Polyolefin Geosynthetics by High-Pressure Differential Scanning Calorimetry
E473 Terminology Relating to Thermal Analysis and Rheology
E537 Test Method for The Thermal Stability of Chemicals by Differential Scanning Calorimetry
E1142 Terminology Relating to Thermophysical Properties
E1782 Test Method for Determining Vapor Pressure by Thermal Analysis
E1858 Test Method for Determining Oxidation Induction Time of Hydrocarbons by Differential Scanning Calorimetry
E2009 Test Methods for Oxidation Onset Temperature of Hydrocarbons by Differential Scanning Calorimetry
E2161 Terminology Relating to Performance Validation in Thermal Analysis

3. Terminology

3.1 *Definitions:*

3.1.1 The technical terms used in this test method are defined in Terminologies E473, E1142, and E2161, including calibration, Celsius, differential scanning calorimetry, high pressure, linearity, oxidative induction time, thermal analysis, and vapor pressure.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *absolute pressure, n*—pressure measured relative to zero pressure corresponding to empty space.

[1] This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.10 on Fundamental, Statistical and Mechanical Properties.

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[2] 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.

3.2.1.1 *Discussion*—

Absolute pressure is atmospheric pressure plus gage pressure.

3.2.2 *atmospheric pressure, n*—the pressure due to the weight of the atmosphere.

3.2.2.1 *Discussion*—

Atmospheric pressure varies with elevation above sea level, acceleration due to gravity and weather conditions. Standard atmospheric pressure is 101.325 kPa.

3.2.3 *barometer, n*—an instrument for measuring atmospheric pressure.

3.2.4 *gage pressure, n*—pressure measured relative to atmospheric pressure.

3.2.4.1 *Discussion*—

Zero gage pressure is equal to atmospheric pressure. Gage pressure is the difference between absolute pressure and atmospheric pressure.

3.2.5 *pressure, n*—the force exerted to a surface per unit area.

3.2.6 *vacuum, n*—pressure less than atmospheric pressure.

4. Summary of Test Method

4.1 The pressure (vacuum) signal generated by a thermal analyzer is compared to a gage whose performance is known and traceable to a national metrology institute. The thermal analyzer may be said to be in conformance if the performance is within established limits. Alternately, the pressure signal may be calibrated using a two-point calibration method.

5. Significance and Use

5.1 Most thermal analysis experiments are conducted under ambient pressure conditions using isothermal or temperature time rate of change conditions where time or temperature is the independent parameter. Some experiments, however, are conducted under reduced or elevated pressure conditions where pressure is an independent experimental parameter (Test Method E537). Oxidation Induction Times (Test Methods D5483, D5885, D6186, and E1858), Oxidation Onset Temperature (Test Method E2009), and the Vapor Pressure (Test Method E1782) are other examples of experiments conducted under elevated or reduced pressure (vacuum) conditions. Since in these cases pressure is an independent variable, the measurement system for this parameter shall be calibrated to ensure interlaboratory reproducibility.

5.2 The dependence of experimental results on pressure is usually logarithmic rather than linear.

6. Apparatus

6.1 *Reference pressure gage* with a range 1.2 times the maximum value to be calibrated readable to within 0.1 % of the full range and performance of which has been verified using standards and procedures traceable to a national metrology institute (such as the National Institute of Standards and Technology (NIST)).

NOTE 1—To ensure an accurate pressure measurement, the reference pressure gage shall be placed as close as practical to the thermal analysis apparatus to be calibrated and connected to the thermal analysis apparatus with large diameter tubing such as 6.3 mm or larger especially for vacuum testing. Ensure that there is no gas flow in the connection (for example, due to leaking) to provide a static pressure measurement.

NOTE 2—Additional information on pressure gages may be found in Practice D5720.

6.2 A *source of pressurized inert gas*, typically nitrogen, with a pressure regulator, capable of adjusting the pressure supplied to the apparatus from zero to 100 % of the gage pressure range to be calibrated, commonly 7 MPa.

NOTE 3—Since the calibration is performed under static flow conditions, the pressurizing gas delivery system to the thermal analysis apparatus should be of small diameter (such as 1.6 mm diameter tubing) for safety considerations.

NOTE 4—Do not use a reactive gas such as oxygen unless all apparatus, tubing and test gage have been cleaned and are rated for oxygen service.

6.3 The thermal analysis apparatus for which the pressure calibration is to be performed.

6.4 *Barometer* capable of measuring atmospheric pressure readable to ±0.01 kPa (0.1 mm Hg).

7. Hazards

7.1 This test poses risks associated with high pressure operation. The thermal analysis apparatus, connecting tubing and measurement gages shall be designed to contain pressures in excess of two times the maximum allowable working pressure. Pressure relief shall be provided at pressures no greater than 1.2 times the maximum allowable working pressure.

8. Preparation of Apparatus

8.1 Assemble the apparatus so that the calibration pressure gage is connected in parallel with the pressure transducer of the apparatus. That is, the instrument transducer and the calibration gage shall see the same static pressure (see Fig. 1). Equilibrate the thermal analysis apparatus pressure container, reference pressure gage and instrument transducer at ambient temperature.