Designation: E2744 – 16

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

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 Methods 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 and Rheology

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.

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

---

[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.

Current edition approved Feb. 15, 2016. Published March 2016. Originally approved in 2010. Last previous edition approved in 2015 as E2744 – 10 (2015). DOI:101520/E2744-16.

[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.

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

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.

9. Calibration

9.1 Perform any pressure signal calibration procedures recommended by the manufacturer of the thermal analyzer as described in the Operator's Manual.

10. Procedure

10.1 Electronic pressure signals associated with thermal analysis apparatus measure gage pressure relative to atmospheric pressure. However, absolute pressure is most often required for thermal analysis experiments. Absolute pressure is the sum of gage pressure and atmospheric pressure. So knowledge of atmospheric pressure is required to obtain absolute pressure.

10.2 Using a laboratory barometer, measure and record the atmospheric pressure (*Patm*) within one hour of the pressure calibration in steps 10.4 – 10.6.

NOTE 5—Should a laboratory barometer be unavailable, local pressure may often be obtained by contacting the local weather service. This approach is not suitable for laboratories operating under negative gage pressure.

10.3 Assemble the instrument to be calibrated, the reference pressure gage and the source of the pressurized gas according to schematic Fig. 1.

10.4 With the thermal analysis exhaust valve open to atmospheric and the source shut-off valve closed (see Fig. 1), set the thermal analysis instrument indicated pressure to zero gage pressure.

10.5 Close the thermal analyzer exhaust valve, open the source of pressurized gas, and slowly increase the pressure regulator until the reference pressure gage reads the maximum pressure to be calibrated (often 7.00 MPa). Close the source valve. Record this value as *P2*.

NOTE 6—Other calibration pressures may be used but shall be reported.

10.6 Record the indicated pressure on the thermal analyzer pressure measuring signal (or gage) as *P3*.

10.7 Calculate the calibration constant (*S*) using Eq 2.

10.8 Using the value of *S* from 10.7, calculate the percent conformity (*C*) using Eq 4 or a table of percent conformity values (see 11.4.1)

11. Calculation

11.1 For the purpose of these procedures, it is assumed that the relationship between observed pressure (*Po*) and the actual pressure (*P*) is linear and governed by Eq 1:

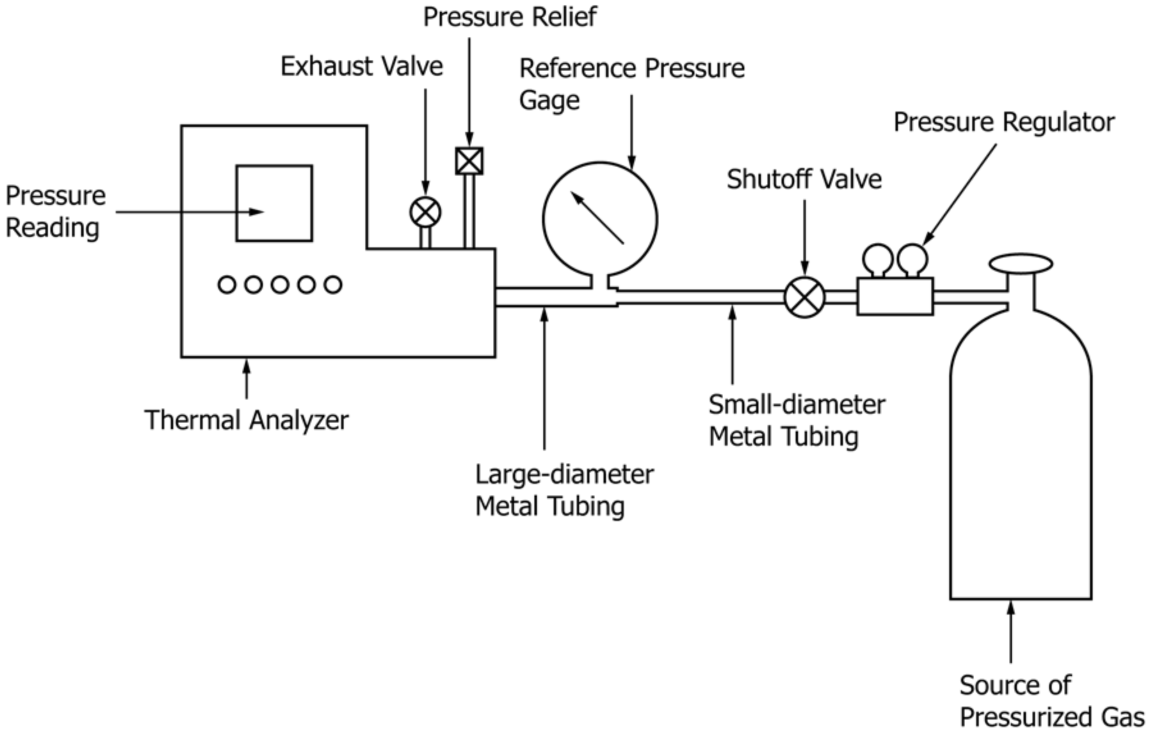

FIG. 1 Schematic Diagram of Apparatus

$$P = Po \times S \quad (1)$$

where:

P = true gage pressure (kPa),
Po = thermal analyzer observed gage pressure (kPa), and
P = calibration constant (nominal value 1.00000).

11.2 The calibration constant *S* is determined by Eq 2:

$$S = \frac{P2}{P3} \quad (2)$$

NOTE 7—When performing this calculation, retain all available decimal places in the measured value and in the value of *S*.

11.3 Using the value of *S* from 11.2, the percent conformity of the pressure measurement of the instrument signal may be calculated by:

$$C = (S - 1.00000) \times 100\,\% \quad (3)$$

11.4 Conformity may be estimated to one significant figure using the following criteria:

11.4.1 If the value of *S* lies:

11.4.1.1 Between 0.9990 and 0.9999 or between 1.0001 and 1.0010, then conformity is better than 0.1 %;

11.4.1.2 Between 0.9900 and 0.9990 or between 1.0010 and 1.0100, then conformity is better than 1 %; and

11.4.1.3 Between 0.9000 and 0.9900 or between 1.1000 and 1.0100, then conformity is better than 10 %.

11.5 Using the determined value of *S*, Eq 1 may be used to calculate true gage pressure (*P*) from an observed signal pressure (*Po*), provided that the measuring gage has been properly "zeroed."

11.6 Absolute pressure (*Pa*) may be obtained from Eq 4:

$$Pa = Patm + P \quad (4)$$

where:

Pa = absolute pressure (kPa), and
$Patm$ = atmospheric pressure (kPa).

12. Report

12.1 Report the following information:

12.2 Model number and description of the thermal analyzer used.

12.3 The value of *S* determined in 10.7 reported to at least four places to the right of the decimal point.

12.4 Conformity as determined in 10.8.

13. Precision and Bias

13.1 An interlaboratory study of pressure signal calibration was conducted in 2009 in which a single organization made 6 duplicated determinations on 5 different instruments for a total of 20 degrees of experimental freedom.

13.2 *Precision:*

13.2.1 Within laboratory variability may be described using the repeatability value (r) obtained by multiplying the repeatability standard deviation by 2.8. The repeatability value estimates the 95 % confidence limit. That is, two results from the same laboratory should be considered suspect (95 % confidence level) if they differ by more than the repeatability value.

13.2.2 The within laboratory repeatability standard deviation obtained for the measurement of pressure was 3.4 kPa. The relative repeatability standard deviation was 0.098 %.

13.2.3 The between laboratory variability may be described using the reproducibility value (R) obtained by multiplying the reproducibility standard deviation by 2.8. The reproducibility value estimates the 95 % confidence limit. This is, results obtained from two different laboratories, operators or apparatus should be considered suspect (at the 95 % confidence level) if they differ by more than the reproducibility value.

13.2.4 The between laboratory reproducibility standard deviation obtained for the measurement of pressure was 6.2 kPa. The relative reproducibility standard deviation was 0.18 %.

13.3 *Bias:*

13.3.1 Bias is the difference between the mean value obtained and an acceptable reference value. This test method reports bias as conformance.

13.3.2 The mean value of pressure measured was 3444.5 kPA gage compared to the reference value of 3447.5 kPa gage. This corresponds to a bias of –3.0 Pa or –0.087 %.

13.4 Bias is the difference between the mean value obtained and an acceptable reference value. This test methods reports bias as conformance.

13.5 The mean slope determined by this test method was $S = 1.00087$. This corresponds to a conformance value of $C = 0.0087$ %.

14. Keywords

14.1 absolute pressure; atmospheric pressure; calibration; gage pressure; pressure; thermal analysis

SUMMARY OF CHANGES

Committee E37 has identified the location of selected changes to this standard since the last issue (E2744 – 10 (2015)) that may impact the use of this standard. (Approved Feb. 15, 2016.)

(1) Revised 10.4 and 10.5.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; http://www.copyright.com/