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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers¹

This standard is issued under the fixed designation E 967; 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 practice covers the temperature calibration of differential thermal analyzers and differential scanning calorimeters over the temperature range from -40 to +2500°C.

1.2 Computer or electronic based instruments, techniques, or data manipulation equivalent to this practice may also be used.

1.3 The values stated in SI units are to be regarded as the 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.* Specific precautionary statements are given in Section 7.

2. Referenced Documents

2.1 ASTM Standards:

E 473 Terminology Relating to Thermal Analysis²

E 968 Practice for Heat Flow Calibration of Differential Scanning Calorimeters²

E 1142 Terminology Relating to Thermophysical Properties²

3. Terminology

3.1 Specific technical terms used in this practice are defined in Terminologies E 473 and E 1142.

4. Summary of Practice

4.1 This practice consists of heating the calibration materials at a controlled rate in a controlled atmosphere through a region of known thermal transition. The difference in heat flow or temperature between the calibration material and a reference material is monitored and continuously recorded. A transition is marked by the absorption of energy by the specimen resulting in a corresponding endothermic peak in the heating curve.

NOTE 1—Heat flow calibrations are sometimes determined in conjunc-

tion with temperature calibration. Some differential scanning calorimeters and quantitative differential thermal analyzers permit both heat flow and temperature calibrations to be obtained from the same experimental procedure.

5. Significance and Use

5.1 Differential scanning calorimeters and differential thermal analyzers are used to determine the transition temperatures of materials. For this information to be meaningful in an absolute sense, temperature calibration of the apparatus or comparison of the resulting data to that of known standard materials is required.

5.2 This practice is useful in calibrating the temperature axis of differential scanning calorimeters and differential thermal analyzers.

6. Apparatus

6.1 Apparatus shall be of either type listed below:

6.1.1 *Differential Scanning Calorimeter (DSC)*, capable of heating a test specimen and a reference material at a controlled rate and of automatically recording the differential heat flow between the sample and the reference material to the required sensitivity and precision.

6.1.2 *Differential Thermal Analyzer (DTA)*, capable of heating a test specimen and reference material at a controlled rate and of automatically recording the differential temperature between sample and reference material both to the required sensitivity and precision.

6.2 *Specimen Pans*, for DSC, composed of clean aluminum or of other high thermal conductivity material. For DTA, specimen cups or tubes composed of clean borosilicate glass, alumina, platinum, aluminum, or quartz may be used. Specimen containers composed of tungsten, graphite or silicon carbide/nitride may be used above 1600°C. The specimen pans or tubes must not react with the specimen and must not melt under the temperatures of the test.

6.3 *Nitrogen*, or other inert purge gas supply.

6.4 *Analytical Balance*, with a capacity greater than 20 mg, capable of weighing to the nearest 0.1 mg.

7. Precautions

7.1 Toxic or corrosive effluents, or both, may be released when heating some material and could be harmful to personnel and to apparatus.

7.2 Some differential thermal analyzers and differential

¹ This practice is under the jurisdiction of ASTM Committee E-37 on Thermal Measurements and is the direct responsibility of Subcommittee E 37.01 on Test Methods and Practices.

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² *Annual Book of ASTM Standards*, Vol 14.02.