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Designation: E 1269 – 99

Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry[1]

This standard is issued under the fixed designation E 1269; 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 covers the determination of specific heat capacity by differential scanning calorimetry.

1.2 This test method is generally applicable to thermally stable solids and liquids.

1.3 The normal operating range of the test is from − 100 to 600°C. The temperature range can be extended, depending upon the instrumentation and specimen holders used.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 Computer or electronic-based instrumentation, techniques, or data treatment equivalent to this test method may be used.

NOTE 1—Users of this test method are expressly advised that all such instruments or techniques may not be equivalent. It is the responsibility of the user of this test method to determine equivalency prior to use.

1.6 *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 9.

2. Referenced Documents

2.1 *ASTM Standards:*

E 473 Terminology Relating to Thermal Analysis[2]

E 967 Practice for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers[2]

E 968 Practice for Heat Flow Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers[2]

E 1142 Terminology Relating to Thermophysical Properties[2]

3. Terminology

3.1 *Definitions*—Technical terms used in this test method are described in Terminologies E 473 and E 1142.

4. Summary of Test Method

4.1 This test method consists of heating the test material at a controlled rate in a controlled atmosphere through the region of interest. The difference in heat flow into the test material and a reference material or blank due to energy changes in the material is continually monitored and recorded.

5. Significance and Use

5.1 Differential scanning calorimetric measurements provide a rapid, simple method for determining specific heat capacities of materials.

5.2 Specific heat capacities are important for reactor and cooling system design purposes, quality control, and research and development.

6. Interferences

6.1 Since milligram quantities of specimen are used, it is essential that specimens are homogeneous and representative.

6.2 The occurrence of chemical changes or mass loss on heating during the measurement may invalidate the test. Therefore, the temperature range and specimen holders should be chosen so as to avoid these processes.

7. Apparatus

7.1 *Differential Scanning Calorimeter (DSC)*—The essential instrumentation required to provide the minimum differential scanning calorimetric capability for this method includes:

7.1.1 *DSC Test Chamber*, composed of the following:

7.1.1.1 *Furnace(s)*, to provide uniform controlled heating (cooling) of a specimen and reference to a constant temperature or at a constant rate within the applicable –100 to 600 °C temperature range of this test method.

7.1.1.2 *Temperature Sensor*, to provide an indication of the specimen temperature to ± 10 mK (0.01 °C).

7.1.1.3 *Differential Sensor*, to detect heat flow difference between the specimen and reference equivalent to 1 µW.

7.1.1.4 A means of sustaining a test chamber environment of inert purge gas at a purge flow rate of 10 to 50 mL/min ± 5 mL/min.

NOTE 2—Typically, 99+ % pure nitrogen, argon, or helium are employed when oxidation in air is a concern. Unless effects of moisture are to be studied, use of dry purge gas is recommended and is essential for operation at subambient temperatures.

[1] This test method is under the jurisdiction of ASTM Committee E-37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Test Methods and Recommended Practices.

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

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7.1.2 *Temperature Controller*, capable of executing a specific temperature program by operating the furnace(s) between selected temperature limits at a rate of temperature change of 10 to 20 °C/min constant to ± 0.1 °C/min or at an isothermal temperature constant to ± 0.1 °C.

7.1.3 *Recording Device*, either digital or analog, capable of recording and displaying any fraction of the heat flow signal (DSC curve) including the signal noise as a function of temperature.

7.1.4 While not required, the user may find useful software to perform the mathematical treatments described in this test method.

7.1.5 *Containers* (pans, crucibles, vials, etc., and lids) that are inert to the specimen and reference materials and which are of suitable structural shape and integrity to contain the specimen and reference in accordance with the specific requirements of this test method.

7.1.6 Cooling capability to hasten cool down from elevated temperatures, to provide constant cooling rates of up to 10 °C/min, to achieve subambient operation, or to sustain an isothermal subambient temperature, or a combination thereof.

7.2 *Balance*, with a capacity of 100 mg or greater to weigh specimens or containers, or both, to± 10 µg.

8. Reagents and Materials

8.1 Specific heat capacity standard: synthetic sapphire disk, 10 to 100 mg.

NOTE 3—Interlaboratory studies indicate that physical forms of the synthetic sapphire other than disks give lower precision and greater bias in the results.

9. Hazards

9.1 *Safety Precautions*—If a specimen is heated to decomposition, toxic or corrosive products may be released.

9.2 *Technical Precautions*:

9.2.1 The same heating rate should be used for both the calibration and specimen runs.

9.2.2 Precision of heating rate, placement of the specimen holder, use of flat specimen holders, and the establishment of equilibrium are essential. Instrument settings should not be adjusted once a specific heat capacity calibration has been performed.

10. Sampling

10.1 Powdered or granular specimens should be mixed prior to sampling and should be sampled by removing portions from various parts of the container. These portions, in turn, should be combined and mixed to ensure a representative specimen for the determinations.

10.2 Liquid specimens may be sampled directly after stirring.

10.3 Solid specimens may be sampled by cutting or slicing with a clean knife or razor blade. Sample uniformity should be ascertained, since segregation within the solid is possible.

NOTE 4—Solid specimens should be so sampled as to maximize contact with the surface of the specimen holder.

10.4 Samples are usually analyzed as received. If some heat or mechanical treatment is applied to the specimen prior to analysis, this treatment should be noted in the report.

11. Calibration

11.1 Specific heat capacity is a quantitative measurement of energy made as a function of temperature. Thus, the instrument used in its measurement must be calibrated in both the temperature and heat flow modes. Since specific heat capacity is not a rapidly changing function of temperature, the instrument's temperature mode is ordinarily calibrated and checked only occasionally. The heat flow information, however, is much more critical and becomes an integral part of the specific heat capacity measurement through the use of a reference material.

11.2 Perform any calibration procedures described by the manufacturer in the operations manual.

11.3 Perform a temperature calibration for the apparatus using Practice E 967.

11.4 Perform a heat flow calibration for the apparatus using Practice E 968.

11.5 *Heat Flow Calibration*:

11.5.1 Synthetic sapphire disk (α-aluminum oxide; alumina) is recommended as a heat flow calibration standard for specific heat capacity measurements. Specific heat capacity values for synthetic sapphire are given in Table 1.

NOTE 5—It is possible to use other standard materials or other physical forms of synthetic sapphire, but their use should be noted in the report. The potential adverse impact of increased interfacial resistance encountered with granular/textured samples may be minimized with the use of a powdered synthetic sapphire standard. It is preferred that the physical form of the sample be similar to that of the standard.

11.5.2 The heat flow calibration may be performed at some regular interval or prior to every specific heat capacity determination or test specimens.

NOTE 6—A frequency of calibration of at least once a day is recommended. Other time intervals may be selected for heat flow calibration but should be noted in the report.

11.5.3 If the heat flow calibration is performed at a regular interval, the calorimetric sensitivity, *E*, may be calculated using the specific heat capacity values for synthetic sapphire given in Table 1 and the following equation:

$$E = [b/(60 \cdot Dst)]\,[Wst \cdot Cp(st) + \Delta W \cdot Cp(c)] \quad (1)$$

Refer to Section 13 for the procedure and Section 14 for the list of symbols.

11.5.4 If the heat flow calibration is performed prior to every specific heat capacity determination, it is unnecessary to calculate the calorimetric sensitivity, *E*. Refer to Section 13 for the procedure.[3]

12. Conditioning

12.1 Specimens and specimen holders for specific heat capacity determinations may be handled in ordinary laboratory environments for screening or qualitative measurements. However, if quantitative data are needed over a wide temperature range, specimen conditioning may be required. Specimens which will be exposed to low temperatures should be protected from moisture. Specimens that will be exposed to very high

[3] The boldface numbers in parentheses refer to the list of references at the end of this standard.

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