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Standard Practice for Description of Thermal Analysis and Rheology Apparatus[1]

This standard is issued under the fixed designation E1953; 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 generic descriptions of apparatus used for thermal analysis or rheometry measurements and its purpose is to achieve uniformity in description of thermal analysis, rheometry, and viscometer instrumentation throughout standard test methods. These descriptions are intended to be used as templates for inclusion in any test method where the thermal analysis instrumentation described herein is cited.

1.2 Each description contains quantifiable instrument performance requirements to be specified for each test method.

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:*[2]

E473 Terminology Relating to Thermal Analysis and Rheology

E1142 Terminology Relating to Thermophysical Properties

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System

3. Terminology

3.1 Technical terms used in this document are found in Terminologies E473 and E1142 and Standard IEEE/ASTM SI 10.

4. Significance and Use

4.1 Section 5 identifies essential instrumentation and accessories required to perform thermal analysis, rheometry, or viscometry for a variety of different instruments. The appropriate generic instrument description should be included in any test method describing use or application of the thermal analysis, rheometry, or viscometry instrumentation described herein.

4.2 Units included in these descriptions are used to identify needed performance criteria and are considered typical. Other units may be used when including these descriptions in a specific test method. Items underlined constitute required inputs specifically established for each test method (for example, sensitivity of temperature sensor).

4.3 Additional components and accessories may be added as needed, with the appropriate performance requirements specified. Items listed in these descriptions but not used in a test method (for example, vacuum system) may be deleted.

5. Apparatus

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

5.1.1 *DSC Test Chamber* composed of:

5.1.1.1 A furnace(s) to provide uniform controlled heating or cooling of a specimen and reference to a constant temperature or at a constant rate within the applicable temperature range of this method.

5.1.1.2 A temperature sensor to provide an indication of the specimen temperature to ± ______ K.

5.1.1.3 Differential sensors to detect a heat flow (power) difference between the specimen and reference with a range of ______ mW and a sensitivity of ± _______ µW.

5.1.1.4 A means of sustaining a *test chamber environment* of ______ at a purge rate of mL/min ± ______ mL/min.

NOTE 1—Typically, ______ % pure nitrogen, argon, or helium is 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.

5.1.2 A *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 ______ K/min constant to ± ______ K/min (list

[1] This practice is under the jurisdiction of 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.

cooling requirements separately if different) or at an isothermal temperature constant to ± ______ K.

5.1.3 A *data collection device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for DSC are heat flow, temperature, and time.

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

5.1.5 *Pressure/Vacuum System* consisting of:

5.1.5.1 A pressure vessel or similar means of sealing the test chamber at any applied pressure within the pressure limits required for this method.

5.1.5.2 A source of pressurized gas or vacuum capable of sustaining a regulated gas pressure in the test chamber of between ______ Pa and ______ Pa.

5.1.5.3 A pressure transducer or similar device to measure the pressure inside the test chamber to ±_______ %, including any temperature dependence of the transducer.

NOTE 2—The link between test chamber and pressure transducer should allow fast pressure equilibration to ensure accurate recording of the pressure above the specimen during testing.

5.1.5.4 A pressure regulator or similar device to adjust the applied pressure in the test chamber to ± ______ % of the desired value.

5.1.5.5 A ballast or similar means to maintain the applied pressure in the test chamber constant to ± ______ Pa or ± ______ %.

5.1.5.6 Valves to control the gas or vacuum environment in the test chamber or to isolate components of the pressure/vacuum system, or both.

5.1.6 Auxiliary instrumentation considered necessary or useful for conducting this method includes:

5.1.6.1 A cooling capability to hasten cool down from elevated temperatures, to provide constant cooling rates, or to sustain an isothermal subambient temperature.

5.1.6.2 A balance to weigh specimens or containers (pans, crucibles, vials, etc.), or both, to ± ______ mg.

5.1.6.3 A means, tool, or device to close, encapsulate, or seal the container of choice.

5.2 *Thermomechanical Analyzer* (TMA)—The essential instrumentation required to provide the minimum thermomechanical analytical or thermodilatometric capability for this method includes:

5.2.1 A rigid specimen holder of inert low expansivity material ______ μm/(m-K) to center the specimen in the furnace and to fix the specimen to mechanical ground.

5.2.2 A rigid (expansion, compression, flexure, tensile, etc.) *probe* of inert low expansivity material ______ μ m/(m-K) which contacts the specimen with an applied compressive or tensile force.

5.2.3 Rigid specimen clamps of inert low expansivity material ______ μm/(m-K) that grip the specimen between the rigid specimen holder and the rigid probe without distortion ______ or slippage______ [for tensile or flexure mode only].

5.2.4 A sensing element linear over a minimum range of______ mm to measure the displacement of the rigid __ probe to ± __ μm resulting from changes in length/height of the specimen.

5.2.5 A weight or force transducer to generate a constant force of ______± ______ [or between _____ and ______± ______] that is applied through the rigid ______probe to the specimen.

5.2.6 A furnace to provide uniform controlled heating or cooling of a specimen to a constant temperature or at a constant rate within the applicable temperature range of this method.

5.2.7 A temperature controller capable of executing a specific temperature program by operating the furnace between selected temperature limits at a rate of temperature change of ______ K/min constant to ± ______ K/min [list cooling requirements separately if different] or at an isothermal temperature constant to ± ______ K.

5.2.8 A temperature sensor to provide an indication of the specimen/furnace temperature to ± ______ K.

5.2.9 A means of sustaining an environment around the specimen of ______ at a purge rate of ______ mL/min ± ______.

NOTE 3—Typically, ______ % pure nitrogen, argon, or helium is 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.

5.2.10 A *data collection device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for TMA are a change in linear dimension, temperature, and time.

5.2.11 Auxiliary instrumentation considered necessary or useful in conducting this method includes:

5.2.11.1 A cooling capability to hasten cool down from elevated temperatures, to provide constant cooling rates or to sustain an isothermal subambient temperature.

5.2.11.2 Micrometer or other *measuring device* to determine specimen dimensions of _____ mm ± _____ mm.

5.2.11.3 A balance with a minimum capacity of __ mg to weigh specimens or clamps, or both, to ± ______ mg.

5.3 *Thermogravimetric Analyzer* (TGA)—The essential instrumentation required to provide the minimum thermogravimetric analytical capability for this method includes:

5.3.1 A thermobalance composed of:

5.3.1.1 A furnace to provide uniform controlled heating or cooling of a specimen to a constant temperature or at a constant rate within the applicable temperature range of this method.

5.3.1.2 A temperature sensor to provide an indication of the specimen/furnace temperature to ± ______ K.

5.3.1.3 A continuously recording balance to measure the specimen mass with a minimum capacity of _____ mg and a sensitivity of ± ______ μg.

5.3.1.4 A means of maintaining the specimen/container under atmospheric control of ______ of ______ % purity at a purge rate of ______ L/min ± _____.

NOTE 4—Excessive purge rates should be avoided as this may introduce interferences due to turbulance effects and temperature gradients.

5.3.2 A temperature controller capable of executing a specific temperature program by operating the furnace between selected temperature limits at a rate of temperature change of ______ K/min constant to within ± ______ K/min (list cooling requirements separately if different) or to an isothermal temperature which is maintained constant to ± ______ K .

5.3.3 A *data collection device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for TGA are mass, temperature, and time.

5.3.4 Containers (pans, crucibles, etc.) that are inert to the specimen and that will remain gravimetrically stable within the temperature limits of this method.

5.3.5 Auxiliary instrumentation considered necessary or useful in conducting this method includes:

5.3.5.1 A *cooling capability* to hasten cool down from elevated temperatures, to provide constant cooling rates, or to sustain an isothermal subambient temperature.

5.4 *Dynamic Mechanical Analyzer (DMA)*, the essential instrumentation required to provide the minimum dynamic mechanical analytical capability for this method, includes:

5.4.1 A *drive motor,* to apply force or displacement to the specimen in a periodic manner capable of frequencies from ____ to _____Hz. This motor may also be capable of providing static force or displacement on the specimen.

5.4.2 A *coupling shaft,* or other means to transmit the force or displacement from the motor to the specimen.

5.4.3 A *clamping system(s),* to fix the specimen between the drive shaft and the stationary clamp(s).

5.4.4 A *position sensor,* to measure the changes in position of the specimen during dynamic motion to ±___ µ m, or

5.4.5 A *force sensor,* to measure the force of ____N developed by the specimen.

5.4.6 A *temperature sensor,* to provide an indication of the specimen temperature to ±___K.

5.4.7 A *furnace,* to provide controlled heating or cooling of a specimen at a constant temperature or at a constant rate within the applicable temperature range of the method.

5.4.8 A *temperature controller,* capable of executing a specific temperature program by operating the furnace between selected temperature limits at a rate of temperature change of ____K/min constant to ±___K (list cooling requirements separately if different) or at an isothermal temperature constant to ±___K.

5.4.9 A *data collection device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for DMA are storage modulus, loss modulus, tan delta, temperature, and time.

5.4.10 Auxiliary instrumentation considered necessary or useful in conducting this method includes:

5.4.10.1 A *cooling capability,* to hasten cool down from elevated temperatures, to provide constant cooling rates, or to sustain an isothermal subambient temperature.

5.4.10.2 *Data analysis capability,* to provide storage modulus, loss modulus, tangent angle delta, or other useful parameters derived from the measure signals.

5.5 *Rheometer*—The essential instrumentation required providing the minimum rheological analytical capabilities for this method include:

5.5.1 A *drive motor*, to apply force or displacement to the specimen in a periodic manner capable of frequencies of oscillation from ___ to ___ rad/s or Hz. This motor may also be capable of providing static force or displacement on the specimen.

5.5.2 A *coupling shaft*, or other means to transmit the force or displacement from the motor to the specimen.

5.5.3 A *geometry or tool* to fix the specimen between the drive shaft and a stationary position.

5.5.4 Either a *force sensor* to measure the force of ___ N developed by the specimen or a *position sensor* to measure the displacement of ___ nm of the test specimen.

5.5.5 A *temperature sensor* to provide an indication of the specimen temperature to ± ___ °C.

5.5.6 6 A *furnace, or heating/cooling element* to provide controlled heating or cooling of a specimen at a constant temperature or at a constant rate within the applicable temperature range of the method.

5.5.7 A *temperature controller*, capable of executing a specific temperature program by operating the furnace or heating/cooling element between selected temperature limits at a rate of temperature change of ___ °C/min constant to ± ___°C/min (list cooling requirements separate if different) or at an isothermal temperature constant to ± ___ °C.

5.5.8 A *stress or strain controller*, capable of executing a specific unidirectional or oscillatory stress or strain program between selected stress or strain limits at a rate of stress or strain change of ___ (units) constant to within ± ____ (units) or at an iso-stress or iso-strain constant to within ± _____ (units).

5.5.9 A *data collection device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for rheology are viscosity, storage modulus, loss modulus, temperature, and time.

5.5.10 Auxiliary instrumentation considered necessary or useful in conducting this method includes:

5.5.10.1 A *cooling capability* to hasten cool down from elevated temperatures, to provide constant cooling rates, or to sustain an isothermal subambient temperature.

5.5.10.2 *Data analysis capability* to provide viscosity, storage modulus, loss modulus, tangent angle delta or other useful parameters derived from the measured signals.

5.6 *Viscometer, Concentric Cylinder Rotational*—The essential instrumentation required providing the minimum rotational viscometer analytical capabilities include:

NOTE 5—A rotational viscometer has many similar components to that of a rheometer described in 5.5 but as a result of its unidirectional force or displacement produces a more limited set of results.

5.6.1 A *drive motor*, to apply a unidirectional torque (force) to the specimen of ____ Nm to ____ Nm constant to ± ____ Nm or unidirectional rotational displacement to the specimen at a rate from ____ rad/s (or r/min) to ___ rad/s (or r/min) constant to ± ___ rad/s (or r/min).

5.6.2 Either a *force sensor* to measure the torque developed by the specimen or a *position sensor* to measure the rotational displacement of the rotational element.

5.6.3 A *coupling shaft*, or other means to transmit the rotational force or displacement from the motor to the specimen.

NOTE 6—It is helpful to have a mark on the shaft to indicate appropriate test fluid level.

5.6.4 A *rotational element, geometry, spindle, or tool* to fix the specimen between the drive shaft and a stationary position.

NOTE 7—Each rotational element typically covers a range of 2 decades of viscosity. The rotational element is selected so that the measured viscosity is between 10 and 90 % of the range of that element.

5.6.5 A *temperature sensor or temperature measuring device* to provide an indication of the specimen temperature of the range of ___ °C to ___ °C to within ± ____ °C.

5.6.6 A *data collection device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for rotational viscosity are torque, rotational speed, temperature, and time.

NOTE 8—Manual observation and recording of data are acceptable.

5.6.7 A *stand*, to support, level, and adjust the height of the drive motor, shaft and rotational element.

5.6.8 A *specimen container*, approximately ___ cm in diameter, ____ cm in depth with a capacity of ___ mL to contain the test specimen during testing.

5.6.9 Auxiliary instrumentation considered necessary or useful in conducting this method includes:

5.6.9.1 *Data analysis capability* to provide viscosity, stress or other useful parameters derived from measured signals.

5.6.9.2 A *cooling capability*, to hasten cool down from elevated temperatures, to provide constant cooling rates, or to sustain an isothermal subambient temperature.

5.6.9.3 A *level* to indicate the vertical plumb of the drive motor, shaft and rotational element.

5.6.9.4 A *guard* to protect the rotational element from mechanical damage.

5.6.9.5 A *temperature bath* to provide a controlled isothermal temperature environment for the specimen or to provide heating or cooling of a specimen at a constant rate within the applicable temperature range of this method.

5.6.9.6 A *temperature controller*, capable of operating the temperature bath at an isothermal temperature over the temperature range of ___ °C to ___ °C constant to ± ____ °C or by executing a specific temperature bath program between ___ °C and ___ °C at a rate of temperature change of ____°C/min constant to ± ___ °C/min.

6. Keywords

6.1 differential scanning calorimeter (DSC); dynamic mechanical analyzer (DMA); rheometer; rheometry; rotational viscometer; thermal analysis; thermogravimetric analyzer (TGA); thermomechanical analyzer (TMA); viscometer; viscometry