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Designation: E1981 – 98 (Reapproved 2020)

Standard Guide for Assessing Thermal Stability of Materials by Methods of Accelerating Rate Calorimetry[1]

This standard is issued under the fixed designation E1981; 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.

INTRODUCTION

This guide is one of several standards being developed by ASTM Committee E27 for determining the physicochemical hazards of chemicals and chemical mixtures. This guide should be used in conjunction with other test methods, as a complete assessment of the hazard potential of chemicals must take into account a number of realistic factors not necessarily considered in this guide. The expression *hazard potential* as used by this committee is defined as the degree of susceptibility of material to ignition or release of energy under varying environmental conditions.

It is the intent of this guide to include any calorimetric device consistent with the principles of adiabatic calorimetry. Device-specific information and specifications are located in appendices to the guide. Any reference to specific devices in the guide are for purposes of illustration or clarity only.

1. Scope

1.1 This guide covers suggested procedures for the operation of a calorimetric device designed to obtain temperature and pressure data as a function of time for systems undergoing a physicochemical change under nearly adiabatic conditions.

1.2 This guide outlines the calculation of thermodynamic parameters from the time, temperature, and pressure data recorded by a calorimetric device.

1.3 The assessment outlined in this guide may be used over a pressure range from full vacuum to the rated pressure of the reaction container and pressure transducer. The temperature range of the calorimeter typically varies from ambient to 500°C, but also may be user specified (see 6.6).

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

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific safety precautions are outlined in Section 7.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E476 Test Method for Thermal Instability of Confined Condensed Phase Systems (Confinement Test) (Withdrawn 2008)[3]

E487 Test Methods for Constant-Temperature Stability of Chemical Materials

E537 Test Method for Thermal Stability of Chemicals by Differential Scanning Calorimetry

E680 Test Method for Drop Weight Impact Sensitivity of Solid-Phase Hazardous Materials

E698 Test Method for Kinetic Parameters for Thermally Unstable Materials Using Differential Scanning Calorimetry and the Flynn/Wall/Ozawa Method

E1231 Practice for Calculation of Hazard Potential Figures of Merit for Thermally Unstable Materials

[1] This guide is under the jurisdiction of ASTM Committee E27 on Hazard Potential of Chemicals and is the direct responsibility of Subcommittee E27.02 on Thermal Stability and Condensed Phases.

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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] The last approved version of this historical standard is referenced on www.astm.org.

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3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *adiabatic calorimeter, n*—an instrument capable of making calorimetric measurements while maintaining a minimal heat loss or gain between the sample and its environment, which is verifiable by the capability to continuously measure the temperature differential between the sample and its surroundings.

3.1.2 *autocatalytic reaction, n*—a chemical reaction in which a product or reaction intermediate functions as a catalyst.

3.1.3 *drift, n*—a gradual unintended increase or decrease in the system (sample container and surroundings) temperature due to limitations in the system calibration, or to changes which occur in the system after calibration.

3.1.4 *final temperature (T_{final}), n*—the observed system temperature at the end of an exotherm, generally at the temperature where the self-heat rate of the reaction has decreased below the operator-defined slope sensitivity threshold.

3.1.5 *heat of reaction (ΔH), n*—the net calculated heat (energy) liberated during an exothermic reaction.

3.1.6 *ideal adiabatic temperature rise (ΔT_{ad}), n*—the temperature rise which would be observed in an exothermic reaction if all of the heat liberated were used to increase the temperature of only the sample. It is conveniently calculated as the product of the observed adiabatic temperature rise, ΔT_{obs}, and the thermal inertia factor, φ.

3.1.7 *observed adiabatic temperature rise (ΔT_{obs}), n*—the observed temperature rise in the system during an exotherm; mathematically, it is equal to the temperature difference between the final temperature and the onset temperature of an exotherm.

3.1.8 *onset temperature (T_{start}), n*—the observed system temperature at the start of an exotherm where the self-heating rate first exceeds the operator-defined slope sensitivity threshold, usually 0.02°C/min; the onset temperature is not a fundamental property of a substance, but is apparatus-dependent, based upon the inherent sensitivity of the calorimetric system.

3.1.9 *self-heating, adj*—any exothermic process which increases the temperature of the system by the self absorption of the liberated heat.

3.1.10 *thermal inertia factor (φ), n*—a correction factor applied to time and temperature differences observed in exothermic reactions in the system (sample and container) under test, which accounts for the sensible heat absorbed by the sample container that otherwise would lead to erroneously low heats of reaction and adiabatic temperature rise, as well as to erroneously high time to maximum rates (TMR's) (see 3.1.12). See also 10.1 for a mathematical formula definition of the thermal inertia factor.

3.1.11 *thermal runaway reaction, n*—a chemical reaction in which the heat generation rate in a system exceeds the heat removal rate of that system.

3.1.12 *time-to-maximum rate (TMR), n*—the amount of time that is needed for a reaction to reach its maximum self-heating rate or pressure rate in a thermal runaway reaction, normally referenced from the time corresponding to the onset temperature, but may also be referenced from any time-temperature point to the time at which the maximum self-heating or pressure rate occurs. The experimentally observed TMR is normally divided by the thermal inertia factor (see 3.1.10) to obtain a more conservative assessment of TMR. (TMR divided by the thermal inertia factor is often referred to as the "φ-corrected" TMR).

4. Summary of Guide

4.1 A sample is placed in a reaction container and positioned in the calorimeter (see Fig. 1).

4.2 The bomb is heated to a user-specified initial temperature and allowed to come to equilibrium, whereupon a search for evidence of an exothermic reaction is undertaken. An exotherm is considered to have occurred when the user-specified rate of temperature rise is first exceeded. If no exotherm is detected, the system temperature is raised a specified increment and the system allowed to equilibrate again. This heat-wait-search cycle is repeated until either an exotherm is detected or the upper temperature limit of the test is reached. If an exotherm is detected, the surroundings are kept at the same temperature as the reaction container, allowing the system to be maintained without heat loss as the temperature of the system increases due to the heat evolved during the exotherm.

4.3 Time, temperature, and pressure data are recorded at specified temperature intervals as a function of time. Additional user-selected parameters may also be recorded or stored.

4.4 The recorded data are used to calculate the time rates of changes of pressure and temperature. These data may also be used to calculate a time-to-maximum rate (as defined in 3.1.12) and to obtain kinetic parameters (**1-4**)[4] for simple, non-autocatalytic exothermic reactions using the equations specified in the vendors' manual (subject to the limitations of 6.5). These data may also be adjusted for the sample- and container-specific heats to calculate an adiabatic temperature rise and heat of reaction.

5. Significance and Use

5.1 The data from this guide seldom, if ever, directly simulate thermal and pressure events in the processing, storage, and shipping of chemicals. However, the data obtained from this guide may be used, with suitable precautions, to predict the thermal and pressure hazards associated with processing, storage, and shipping of a chemical or mixture of chemicals after appropriate scaling of the data. This has been addressed in the literature (**1-4**) but is beyond the scope of this guide.

5.2 This guide is suitable, under the proper conditions, for the investigation of the effects of catalyst, inhibitors, initiators,

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

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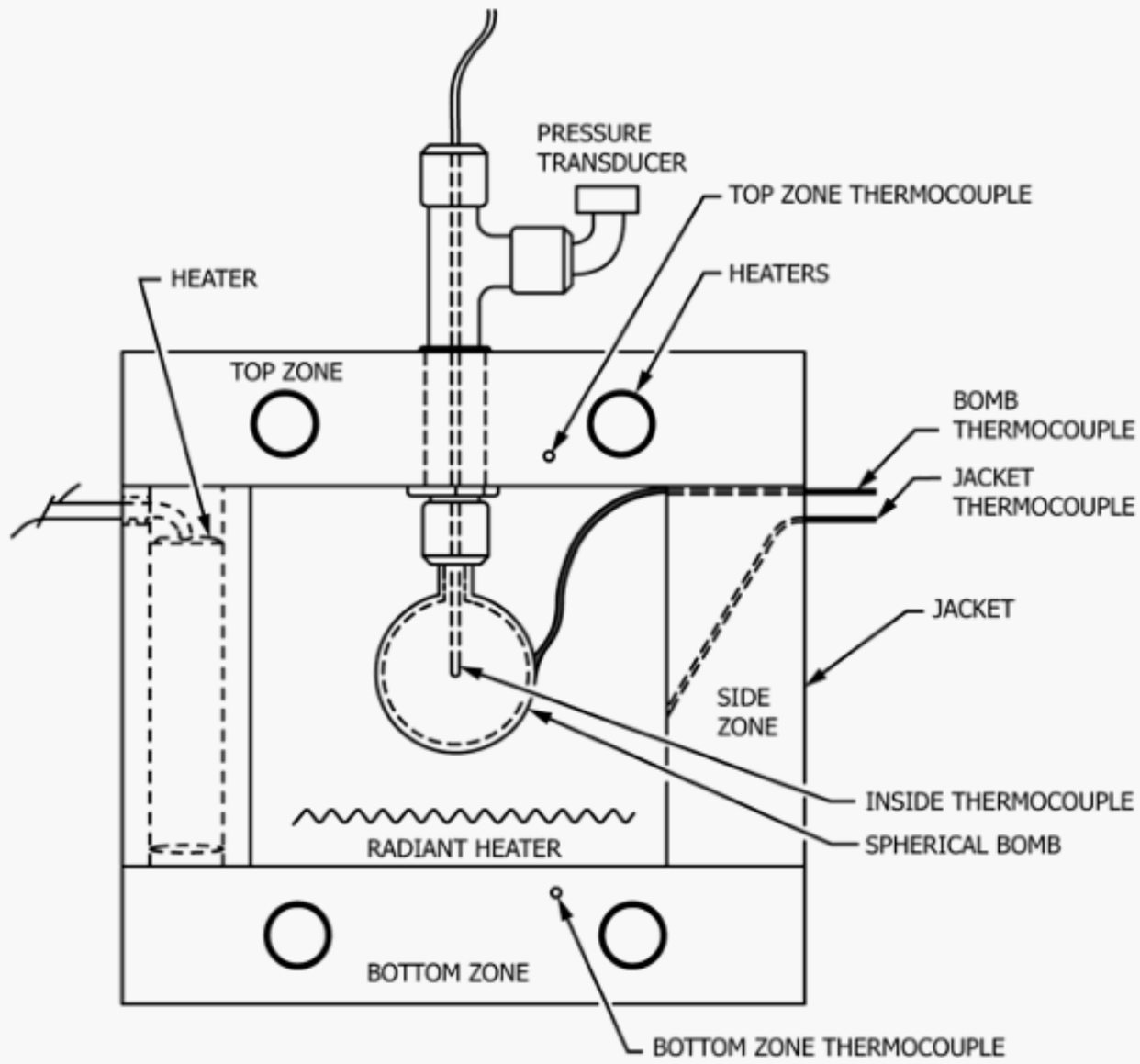

FIG. 1 Example Calorimeter and Reaction Container

reaction atmospheres, materials of construction, or, if available, agitation (see 6.1.2).

5.3 Interpretation of the time-temperature or time-pressure data may be possible for relatively simple systems through the use of suitable temperature-dependent kinetic theories such as the Arrhenius and Absolute Reaction Rate theories (**5**, **6**).

6. Limitations

6.1 This guide requires good heat transfer within the sample and between the sample and the container and, therefore, is subject to the following limitations:

6.1.1 Solid samples or systems where heat transfer could become rate-limiting may not yield quantitatively reliable or consistent results, and

6.1.2 Heterogeneous systems may not give meaningful results. A qualitative indication of change in reaction rate may be obtained by (optional) agitation, but the observed reaction rates may be strongly dependent on the rate and efficiency of the agitation. Loss of agitation may also affect observed reaction rates.

6.2 Accurate tracking of very high or very low self-heat rates may not be quantitatively reliable and is equipment dependent.

6.3 Endothermic reactions can be observed but generally are not quantitatively measured.

6.4 The determination of enthalpies of reaction is based on an accurate knowledge of the (temperature-dependent) heat capacities of the reactants, products, and container. The calculation is also dependent on the temperature tracking accuracy of the system (see 6.2).

6.5 The use of the equations specified for the determination of kinetic parameters (see, for example, Appendix X1) may not be suitable in many instances, especially when multiple reactions are involved.

6.6 Data may be obtained in the temperature range consistent with the calorimeter's specifications and at pressures up to those consistent with the limitation of the pressure transducer or the material of construction of the bomb.

6.7 Modifications to the calorimeter can significantly alter the performance of the instrument. It is the user's responsibility to assure that modifications do not alter the precision or accuracy of the instrument.

6.8 If the thermal inertia (φ) factor for the experiment differs significantly from that of the system it is intended to simulate, any reaction mechanism observed experimentally may not be the same as the true reaction mechanism that exists in the system being simulated.

6.9 In the determination of kinetic parameters, the possibility of autocatalytic reaction mechanisms must be considered.

7. Hazards

7.1 The thermal stability characteristics, impact characteristics, (see Test Methods E476, E487, E537, E680, and E698, Practice E1231, and Ref (**7**)), or friction sensitivity characteristics of the sample, or a combination thereof, should be assessed, as it is often necessary to grind (see Note 1) or compact the sample prior to or during loading into the sample container. Additional physical properties of the sample may also need to be determined, such as sensitivity to electrostatic discharge.

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NOTE 1—Caution should be used in grinding sample materials, as polymorphic changes can occur, thus altering the nature of the sample.

7.2 If the device incorporates a pressure-relief device, it should be periodically inspected for possible corrosion or physical damage, which may result in improper operation.

7.3 Operation of the relief device or rupture of the bomb may result in the release of toxic or noxious fumes, which may escape into the immediate operating area. The calorimeter, therefore, should be properly vented.

7.4 When venting the sample container at the end of the test, suitable precautions should be taken prior to lifting the top cover of the calorimeter in order to prevent exposure of the operator to a potentially highly pressurized container capable of rupture without warning.

7.5 Bombs and transducer lines may become plugged, preventing normal operation of any relief device or vent valve. Therefore, exercise caution and use appropriate personal protective equipment and shielding devices prior to attempts to relieve the pressure. Depressurization and subsequent opening of the sample container at the end of the test should be performed in a safe manner, taking into consideration potential, unanticipated pressure releases or exposure to the operator, or both.

7.6 The toxicity of the contents of the sample container, especially after reaction, should be considered and handled in a manner consistent with local safety and regulatory procedures.

7.7 Material incompatibilities, including that with the silicone oil or other fluids in the pressure transducer or lines, should be considered in any test.

7.8 The mass of the sample and total energy release potential (see Test Methods E476, E487, E537, E680, and E698, Practice E1231, and Ref (**7**)) should always be sufficiently limited so as to minimize the potential for rupture of the sample container due to overpressurization.

7.9 Any safety interlock on the device should not be defeated.

8. Apparatus

8.1 The equipment used in this guide shall be capable of measuring and recording changes of temperature and pressure occurring within a sample (placed inside a suitable container) as a function of either time or temperature, or both.

8.2 The accelerating rate calorimetric device may be purchased or custom built to various degrees of precision and sensitivity. The basic components of an apparatus suitable for this guide include:

8.2.1 Calorimeter,

8.2.2 Sample (reaction) container,

8.2.3 Programmable temperature controller,

8.2.4 Heating unit, and

8.2.5 Temperature and pressure measuring and recording devices.

8.3 Optional components include provision for stirring of the sample and automatic operation (data collection and storage) of the apparatus after test initialization.

8.4 The apparatus shall employ the principles of adiabatic calorimetry to minimize heat loss from the reaction container to the surrounding environment.

8.5 The calorimeter shall be adequately shielded and vented in order to protect the operator from any sample container rupture or detonation within the calorimeter and from any resulting effluent. (see Section 7).

9. Procedure

9.1 *Calibration:*

9.1.1 An instrument calibration should be carried out on a regular basis or whenever a major change has been made to the system (thermocouple or heater replacement, rupture of the sample container, unacceptable drift, and so forth) and should include the range of temperature used in testing substances.

9.1.2 An instrument calibration is best carried out using an empty, clean, light-weight sample container.

9.1.3 A number of compounds have been selected as suitable for use as system performance verification materials. As an example, Appendix X1 contains a summary of the results of round robin testing for these compounds using an accelerating rate calorimeter. Other suitable devices may use the same or different calibration compounds.

9.2 *Operating Procedure:*

9.2.1 The sample to be tested is loaded into a sample container. Sample containers may be reused; however, it is the responsibility of the user to assure that the containers are properly cleaned prior to reuse. If stirring is to be carried out, a suitably equipped sample container must be used.

9.2.2 The general procedure described in the operating manual corresponding to the particular device being used should be followed.

9.2.3 After the test is completed and the system temperature has returned to ambient conditions, the sample container is removed from the calorimeter (see Section 7) and disposed of in a manner consistent with local safety and regulatory procedures.

9.2.4 It is often useful to record the post-cooled system pressure and temperature of the sample container in order to determine the amount of non-condensable gases evolved.

9.2.5 It is often useful to record temperature and pressure data throughout the entire test, instead of just during an exotherm, as further elucidation of the physicochemical process may be realized.

9.2.6 Further, it is often useful to collect and analyze the head space gases and weigh the residues at the end of the test.

10. Calculation

10.1 The thermal inertial factor is calculated as follows:

$$\varphi = \frac{\Delta T_{ad}}{\Delta T_{obs}} = \frac{(M_S C_S + M_b C_b)}{(M_s C_s)} \quad (1)$$

where:

M_s = mass of sample,
M_b = mass of bomb,
C_s = specific heat capacity of sample, and
C_b = specific heat capacity of bomb.

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This equation does not explicitly allow for temperature-dependent specific heat capacities. Normally, an average value of specific heat capacity is assumed to be constant over the temperature range of interest.

10.2 The observed adiabatic temperature rise is calculated as follows:

$$\Delta T_{obs} = (T_{final} - T_{start}) \quad (2)$$

where:

T_{final} = final temperature of the exotherm, and
T_{start} = onset temperature of the exotherm.

10.3 The ideal adiabatic temperature rise is calculated as follows:

$$\Delta T_{ad} = (\Delta T_{obs})(\varphi) \quad (3)$$

10.4 The heat of reaction per unit mass is calculated as follows:

$$\Delta H = (\Delta T_{ad})(C_s) \quad (4)$$

where:

C_s = specific heat capacity of sample.

The calculation of the heat of reaction is directly proportional to the sample specific heat capacity. It is important that it be accurately determined and its temperature dependence assessed.

10.5 *Calculation of Reaction Rate and Activation Energy Parameters*—Specific information concerning the theoretical interpretation of various temperature-dependent kinetic theories may be found in Refs (**5**, **6**).

11. Report

11.1 Report the following information:

11.1.1 Bomb type,

11.1.2 Atmosphere in the head space,

11.1.3 Bomb mass,

11.1.4 Sample mass,

11.1.5 Volume of sample and container,

11.1.6 Heat capacity of the bomb and the sample,

11.1.7 Start temperature,

11.1.8 Heat step,

11.1.9 Wait time,

11.1.10 Search time,

11.1.11 Isothermal wait time, if any,

11.1.12 End temperature,

11.1.13 Calorimeter exothermic rate threshold and stirring rate, if available,

11.1.14 Plots of various parameters (for example, see Appendix X1), and

11.1.15 Data for all observed exotherms, including:

11.1.15.1 Starting and ending temperatures,

11.1.15.2 Time-to-maximum rate,

11.1.15.3 Maximum observed heat rate,

11.1.15.4 Calculated adiabatic temperature rise (as defined in 3.1.6), and

11.1.15.5 Residual sample mass and pressure.

12. Precision and Bias

12.1 Because a guide does not generate a test result, no precision or bias statement is required. However, typical test results for calibration compounds may be found in the Appendix X1.

13. Keywords

13.1 calorimetry; thermal hazard; thermal stability

APPENDIX

(Nonmandatory Information)

X1. EXAMPLES OF TYPICAL DATA GENERATED BY AN ACCELERATING RATE CALORIMETER TEST

X1.1 Experimental Conditions of Chemical Evaluation Systems

X1.1.1 *Di-tert-Butyl Peroxide (DTBP):*

X1.1.1.1 *Bomb*—titanium, 1.0-in. outside diameter sphere, 0.032-in. wall.

X1.1.1.2 *Volume*—5 mL.

X1.1.1.3 *Headspace*—air.

X1.1.1.4 *Start Temperature*—80°C.

X1.1.1.5 *End Temperature*—250°C.

X1.1.1.6 *Step Temperature*—5°C.

X1.1.1.7 *Wait Time*—15 min.

X1.1.1.8 *Threshold*—0.02°C per min.

X1.1.1.9 *Material*—20 % by weight DTBP in toluene.

X1.1.1.10 Round-robin test data (self-heat rate versus reciprocal absolute temperature) for the system described in X1.1.1 is shown in Fig. X1.1.

X1.1.2 *Azobisisobutyronitrile (AIBN):*

X1.1.2.1 *Bomb*—Hastelloy C, 1.0-in. outside diameter sphere, 0.032-in. wall.

X1.1.2.2 *Volume*—5 mL.

X1.1.2.3 *Headspace*—air.

X1.1.2.4 *Start Temperature*—50°C.

X1.1.2.5 *End Temperature*—175°C.

X1.1.2.6 *Step Temperature*—5°C.

X1.1.2.7 *Wait Time*—15 min.

X1.1.2.8 *Threshold*—0.02°C per min.

X1.1.2.9 *Material*—12 % by weight AIBN in dichloromethane.

X1.1.2.10 Round-robin data (self-heat rate versus reciprocal absolute temperature) for the system described in X1.1.2 are shown in Fig. X1.2.

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X1.1.3 *Methanol + Acetic Anhydride:*

X1.1.3.1 *Bomb*—Hastelloy C, 1.0-in. outside diameter sphere, 0.032-in. wall.

X1.1.3.2 *Volume*—4.65 mL.

X1.1.3.3 *Headspace*—air.

X1.1.3.4 *Start Temperature*—30°C.

X1.1.3.5 *End Temperature*—300°C.

X1.1.3.6 *Step Temperature*—5°C.

X1.1.3.7 *Wait Time*—15 min.

X1.1.3.8 *Threshold*—0.02°C per min.

X1.1.3.9 *Material*—The bomb is first loaded with 2.5 mL acetic anhydride. At 30°C, the instrument is put in exotherm mode with the instrument enabled to record data during the methanol addition step, and 2.15 mL methanol is added in one shot.

NOTE X1.1—Shot addition is a non-standard sample addition method, and should only be used with adequate safety precautions to protect the operator from possible exposure to reacting substances.

X1.1.3.10 Round-robin test results (self-heat rate versus reciprocal absolute temperature) for the system described in X1.1.3 are shown in Fig. X1.3.

X1.2 After the completion of a test, the following plots may be useful:

X1.2.1 Log self-heat rate (°C per min) versus reciprocal absolute temperature (scaled in 1 per K).

X1.2.2 Temperature (°C per min) versus time (min).

X1.2.3 Pressure (psi) versus time (min).

X1.2.4 Log pressure (psi) versus reciprocal absolute temperature (1 per K).

X1.2.5 Log pressure rate (psi per min) versus reciprocal absolute temperature (1 per K).

X1.2.6 Log temperature (°C) versus log time to maximum rate (days, hours, minutes, seconds).

X1.2.7 Pseudo rate constant (K*) versus reciprocal absolute temperature (1 per K).

X1.2.8 Time-to-maximum rate versus temperature (°C); see 3.1.12.

X1.2.9 Pressure (psi) versus temperature (°C).

X1.2.10 Log pressure rate (psi per min) versus log temperature rate (°C per min).

X1.2.11 Activation energy (kcal per mol) versus temperature (°C).

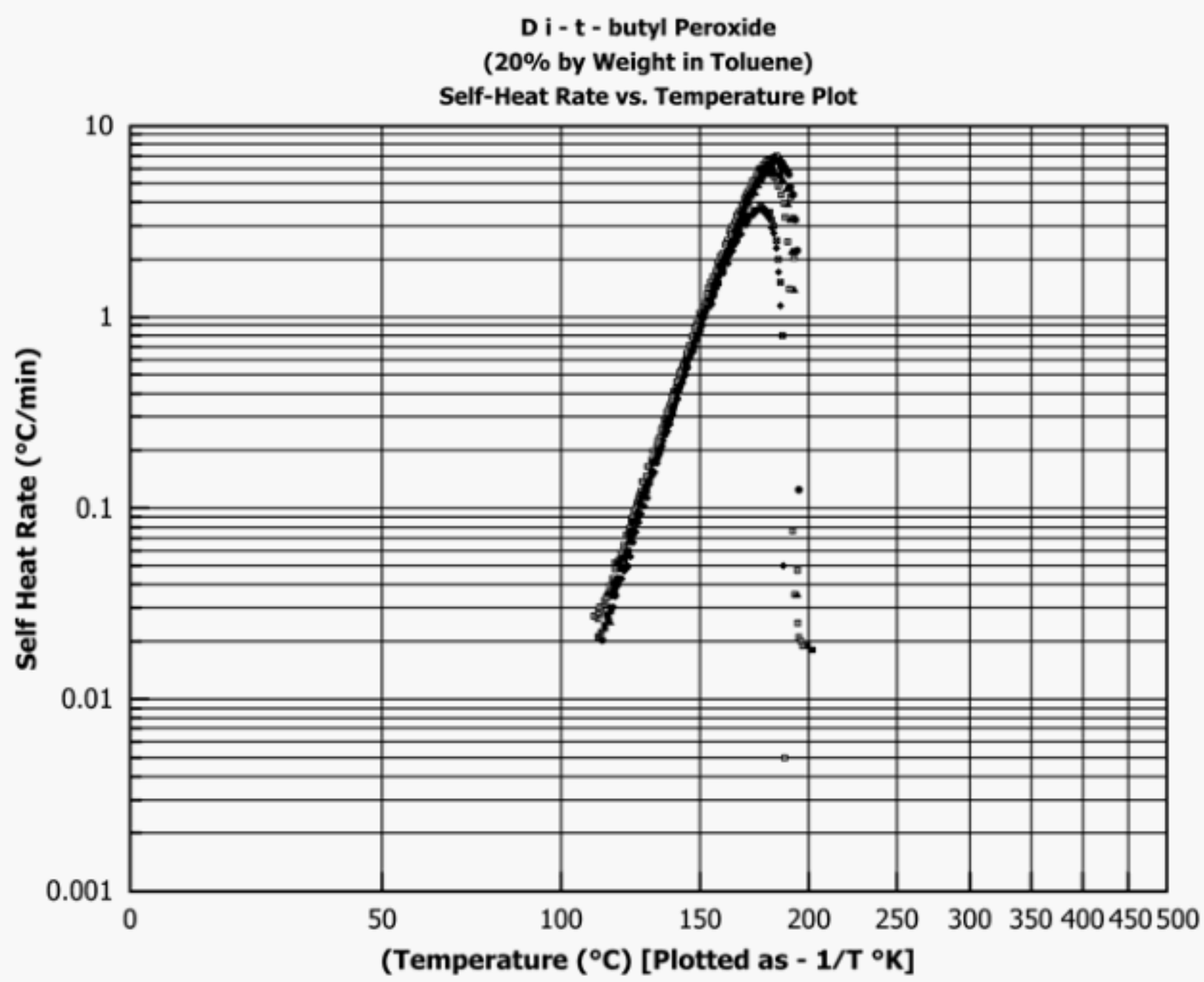

FIG. X1.1 Di-t-Butyl Peroxide (20 Weight Percent in Toluene) Self-Heat Rate versus Temperature Plot

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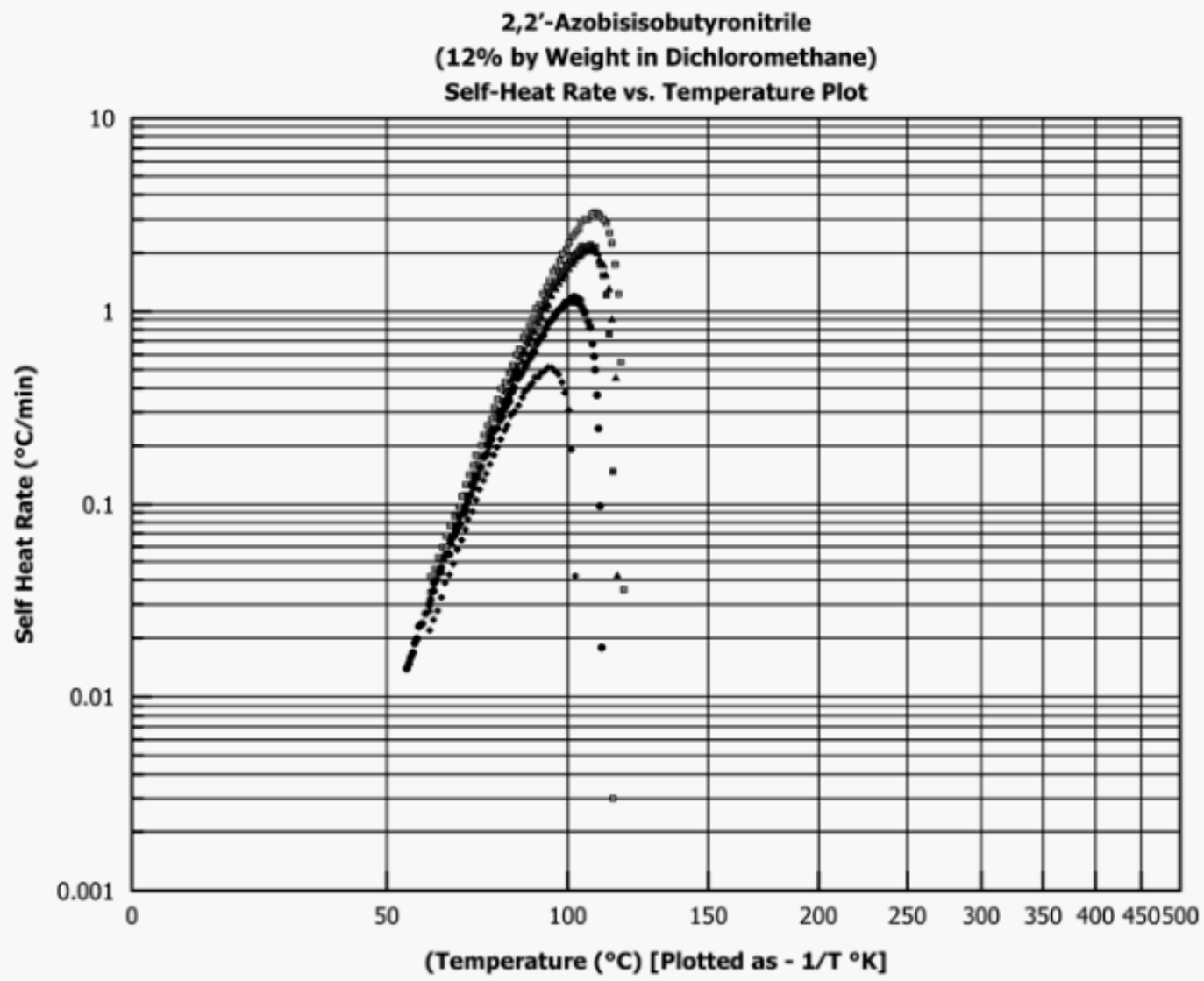

FIG. X1.2 2,2'-Azobisisobutyronitrile (12 Weight Percent in Dichloromethane) Self-Heat Rate versus Temperature Plot

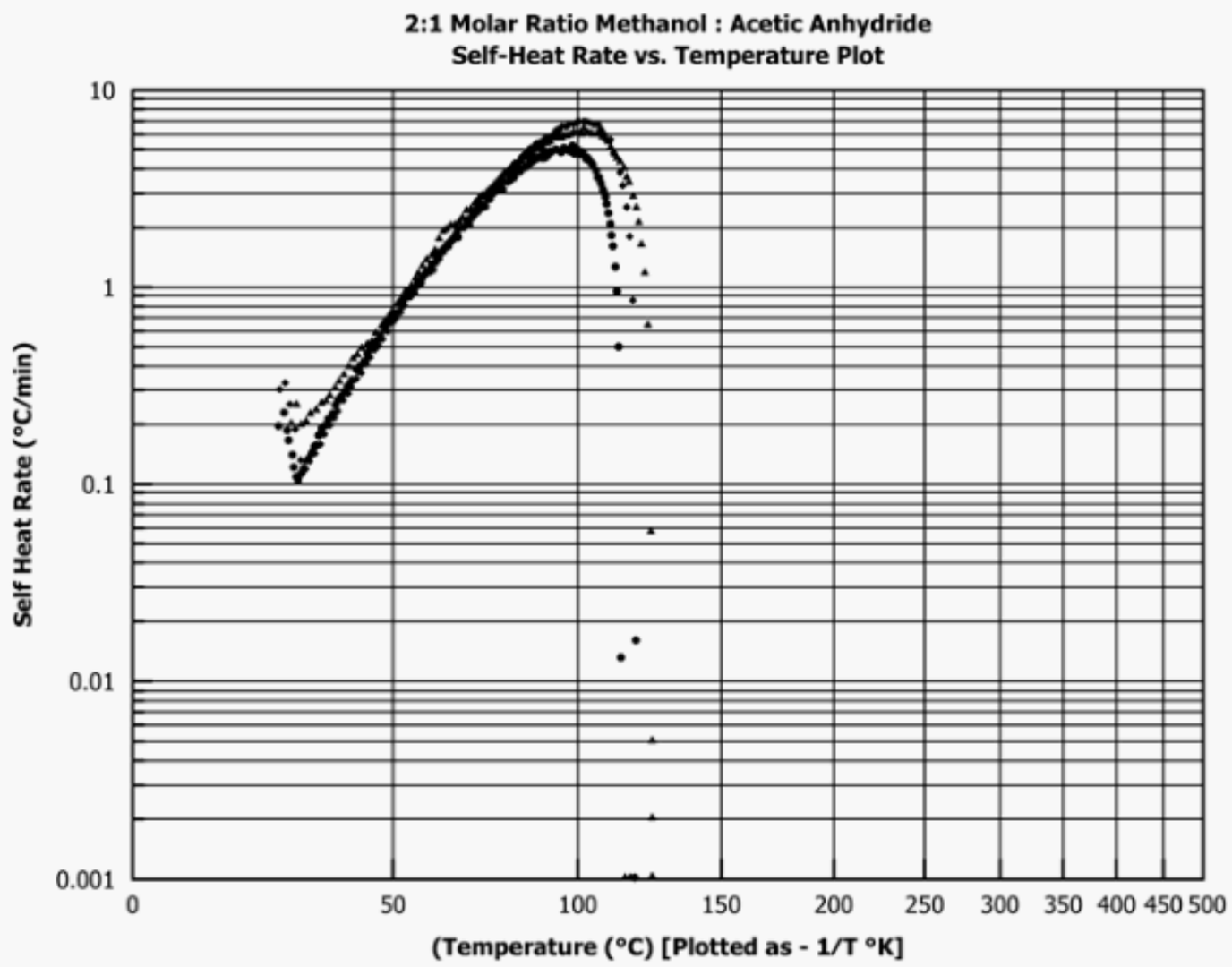

FIG. X1.3 2 to 1 Molar Ratio—Methanol to Acetic Anhydride Self-Heat Rate versus Temperature Plot

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