



Designation: E 476 – 87 (Reapproved 2001)

Standard Test Method for Thermal Instability of Confined Condensed Phase Systems (Confinement Test)¹

This standard is issued under the fixed designation E 476; 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 test method is one of several methods developed by Committee E-27 for determining the hazards of chemicals. This test method is to be used in conjunction with other tests to characterize the hazard potential of chemicals.

1. Scope

1.1 This test method² is designed to determine the temperature at which a chemical or mixture of chemicals, confined initially as a solid or liquid in air or other controlled atmosphere under normal laboratory conditions, will start a reaction, generating appreciable heat when subjected to a programmed temperature increase. This test method is also designed to measure the magnitude and rate of heat generation.

1.2 This test method is for use with condensed phases.

1.3 This test method can be used over a temperature range from 0 to 500°C, and a pressure range of 0 to 5000 psi.

1.4 As with any thermal stability test, proper safety precautions should be instituted to protect personnel. See also Section 6.

1.5 Limitations:

1.5.1 The threshold temperature determined by this method may be higher than one determined by heating at a lesser rate.

1.5.2 Samples of the same material having different thermal histories may have different threshold temperatures.

1.6 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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.*

¹ This test method is under the jurisdiction of Committee E27 on Hazard Potential of Chemicals and is the direct responsibility of Subcommittee E 27.02 on Thermal Stability.

Current edition approved Sept. 25, 1987. Published November 1987. Originally published as E 476 – 73. Last previous edition E 476 – 73 (1979)².

² This test method is a modification of the Thermal Stability Test recommended by the Interagency Chemical Rocket Propulsion Group, published by the Chemical Propulsion Information Agency in May, 1964, and is the responsibility of E 27.02 on Thermal Stability.

2. Terminology

2.1 *threshold temperature*—temperature on the ΔT versus T curve (see Fig. 2) where the slope changes in the direction indicating an exothermic reaction, that is, the sample is beginning to self-heat.

3. Summary of Method

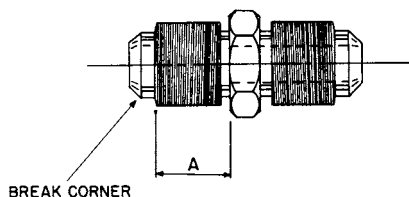
3.1 The sample is confined in a specially designed vessel equipped with a shielded thermocouple. The test assembly is put into a bath and equilibrated, usually at room temperature. The bath is then heated at a constant temperature rise rate. The differential temperature (sample temperature minus bath temperature) in the vessel is recorded versus bath temperature. Heating is continued until the diaphragm bursts or the upper temperature limit is reached. The differential temperature curve is then analyzed to determine the threshold temperature for initiation of measurable reaction as indicated by an exothermic temperature rise.

4. Significance and Use

4.1 The threshold temperature measured by this test method is an indication of the thermal instability of a chemical or mixture of chemicals, qualitatively expressed by the temperature rise. There is a potential hazard whenever the temperature of the chemical exceeds the threshold temperature unless proper design safeguards are utilized. This does not imply that temperatures lower than the threshold temperature are safe. Since this test is not an adiabatic type and does not indicate the effect of mass or time, other testing would be needed to characterize the use or storage of the chemical at lower temperatures.

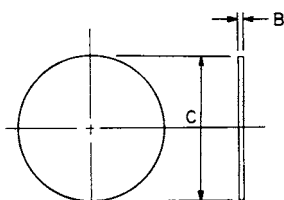
4.2 Because of rate and mass dependent factors, failure to find evidence of an exothermic reaction does not ensure thermal stability unless substantiated by other test methods.

MODIFY ONE END OF 1/4" AN TUBE UNION AS SHOWN
(PARKER 4HBTX-SS OR EQUIVALENT)

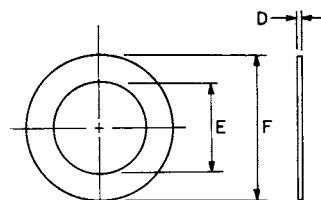


THERMAL STABILITY BOMB CONNECTOR

TABLE OF DIMENSIONS					
	IN.		MM		
	DIMENSION	TOLERANCE	DIMENSION	TOLERANCE	
A	0.420	0.005	10.67	0.13	
B	0.003	—	0.08	—	
C	0.363	+0.002 -0.000	9.20	+0.05 -0.00	
D	0.010	—	0.25	—	
E	0.219	0.015	5.52	0.38	
F	0.362	0.015	9.19	0.38	



BURST DIAPHRAGM
304 STAINLESS STEEL



GASKET
250 ALUMINUM

FIG. 1 Connector, Gasket, and Burst Diaphragm

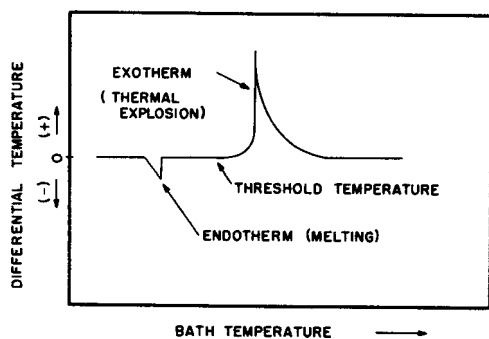


FIG. 2 Idealized Thermogram

5. Apparatus

5.1 Sample Container—A diagram of a suggested test cell assembly is shown in Fig. 3 and an engineering drawing is shown in Fig. 4. The assembly shall consist of the following parts: basic test cell, sample thermocouple, compression fitting, sealing ring, burst diaphragm, modified Army-Navy specification union (AN union), vent tube and flare fitting. Detailed dimensions of all parts are given in Fig. 4 with the exception of those parts readily available from manufacturers. The parts are listed in Table 1. The internal volume of the assembled test cell is approximately 1.1 mL.^{3,4}

5.2 Instrumentation:

5.2.1 One recorder is used for recording the difference between the sample temperature and the bath temperature as a function of bath temperature. A suitable recorder for this test is a standard 8.5 by 11 in. X-Y plotter. Two recorders are required if pressure versus bath temperature is monitored. Dual-pen plotters are suitable, provided the temperature pens do not interact at any critical junction of the reaction.

5.2.2 The maximum reaction rates that can be followed using the recommended instrumentation are limited by the writing speeds of the mechanical writing recorders. When these are calibrated as described in Section 8, rates of reaction producing temperature changes of 5°C per s can be determined. Certain reactions may cause temperature changes in excess of these. If more exact resolution for rapid reactions is desired, it is necessary to use a recording oscillograph in place of the X-Y recorder.

5.3 Low-Range Heating Bath—For temperatures from 0 to 370°C the bath may be a conventional 2-L silicon oil⁵ unit with heaters (1800 W), stirring motor, and temperature programmer. The bath container shall be metal with strip heaters on the outside. The bath shall be well insulated. A cooling coil shall be wrapped around the container to reduce the time lost between tests (optional). The coolant shall be tap water. The nominal heating rate for the bath shall be 8 to 10°C per min.

⁴ This revised test method, when used with its provision for optional pressure measurement, is essentially similar to earlier versions of this in which pressure measurement was an integral part.

⁵ Dow Corning 710 has been found satisfactory for this purpose.

³ Apparatus of similar designs with a total volume of 1 to 10 mL can be used.