NOTICE: This standard has either been superseded and replaced by a new version or discontinued. Contact ASTM International (www.astm.org) for the latest information.

Designation: E 771 – 90 (Reapproved 1996)

Standard Test Method for Spontaneous Heating Tendency of Materials[1]

This standard is issued under the fixed designation E 771; 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 a small-scale laboratory procedure to determine the self heating tendency of liquid and solid chemicals by exposure to elevated temperatures in air in a controlled semi-adiabatic system.

1.2 *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. Terminology

2.1 *Definitions:*

2.1.1 *spontaneous heating or self heating*, *n*—an exothermic reaction of a material due to slow or incomplete reaction that results in a temperature rise above that of its surroundings.

2.1.2 *spontaneous heating temperature or self-heating temperature*, *n*—the lowest temperature at which spontaneous or self heating occurs under the specified test conditions. This temperature refers to a much earlier stage of reaction than that associated with the autoignition temperature of the material.

3. Summary of Test Method

3.1 A small quantity of the test material held within a loosely packed inert material is heated in a thermostatically controlled chamber and the sample temperature is monitored to determine the temperature rise due to exothermic reaction. Bath temperature, sample quantity, and particle size of solids are varied to obtain the relative self-heating temperature of the material.

4. Significance and Use

4.1 This test method is applicable to solid and liquid materials and provides a means of accelerating the tendency of a material toward spontaneous heating which may eventually lead to a fire.

4.2 Tests at temperatures covering the range expected in manufacturing processes or material usage can be of considerable value in determining safe operating conditions.

4.3 Exothermic reaction under test conditions is a positive indication of spontaneous heating tendencies of a material. Negative test results indicate the absence of detectable spontaneous heating behavior under the experimental conditions imposed, but should not be regarded as conclusive for all conditions, particularly those which may be considered adiabatic.

4.4 The spontaneous heating behavior of a material is affected by such factors as available surface area, availability of oxygen to the test specimen, humidity, sample moisture content, packing density, the test temperature, and loss of exothermic heat to the surroundings.

5. Apparatus

5.1 *General*—The complete apparatus (see Fig. 1), consists of a test chamber with a sample well and auxiliary equipment including a heater and temperature control system, supplementary electronic bath temperature controller, high-temperature limit switch, motor stirrer, multichannel recorder, thermocouples, and controlled air supply.

5.1.1 *Test Chamber*—A double-walled insulated container, capable of holding an oil bath at temperatures ranging to 250°C, an immersed sample well, and provision for admission of air at bath temperature to the sample well, will satisfy the needs of this test. A hood is recommended to facilitate removal of noxious products of combustion.

5.1.1.1 The recommended test chamber should be constructed of double-walled stainless steel sheeting or other suitable metal. An 8-in. (203-mm) diameter stainless steel beaker can be used as the inner container and a 9-in. (229-mm) diameter stainless steel beaker can serve as the outer wall with the bottom cut away to conform in height to the inner container and to expose the bottom of the inner container to the heating surface. The annular space between the inner and outer wall is filled with insulating material and assures an integral tight-fitting unit.

5.1.1.2 A 12-ft (3.66-m) coil of ¼-in. (6.35-mm) copper tubing is attached to the bottom of the test sample well by metal fittings to provide a supply of heated air to the test sample. The well and coil are immersed in heat-transfer oil having a flash point well above the maximum operating temperature of the bath. The sample well in the test chamber is supported by the coil and the chamber cover.

5.1.1.3 The test sample well is constructed of a 6-in. (152 mm) length of 3-in. (76-mm) diameter stainless steel pipe of

[1] This test method is under the jurisdiction of ASTM Committee E-27 on Hazard Potential of Chemicals and is the direct responsibility of Subcommittee E27.04 on Flammability and Ignitability of Chemicals.

Current edition approved May 25, 1990. Published July 1990. Originally published as E 771 – 81. Last previous edition E 771 – 81.

Copyright © ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, United States.

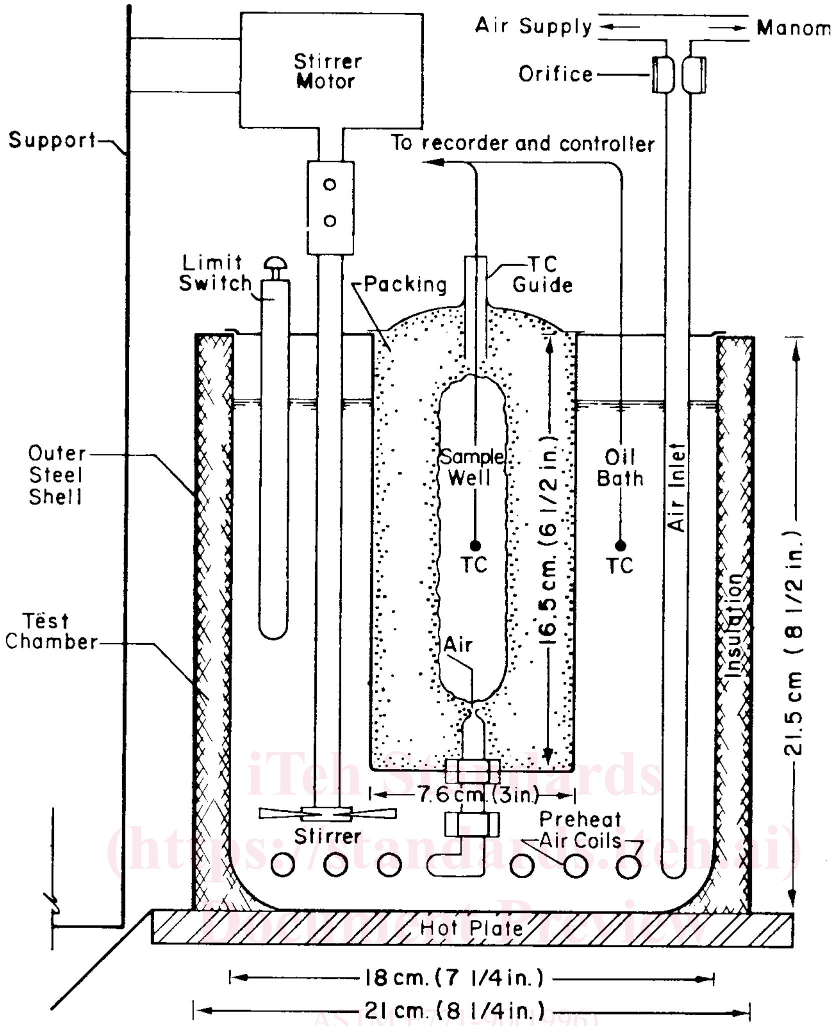

FIG. 1 Spontaneous Heating Apparatus

iTeh Standards
(https://standards.iteh.ai)
Document Preview
ASTM E771-90(1996)
https://standards.iteh.ai/catalog/standards/sist/9f225deb-1513-49d8-b72f-55bb60449c7b/astm-e771-901996

approximately 0.035-in. (0.88-mm) thickness with a welded bottom perforated and fitted to admit air from the coil. The fittings are constructed so that the air is admitted centrally about ½ in. (12.7 mm) above the bottom of the well.

5.1.1.4 The cover of the annular space containing the oil bath support a temperature limit switch and has feed-through facilities for air admission, thermocouples to measure the bath temperature, and a stirrer rod. An 8-in. (203-mm) stainless steel beaker cover may be used for this purpose. The cover of the test sample holder has feed through facilities for thermocouples measuring the sample temperature.

5.1.2 *Controllable Hot Plate*, having a minimum surface area of 81 in.^{2} (523 cm^2) is used to heat the bath.[2]

5.1.2.1 A separate power source from the supplementary controller is wired to the hot plate bypassing the thermostatted potentiometer.

5.1.3 *Supplementary Circuit*, provides power to the bath heater when the sample temperature registers 2 to 3°C higher than the bath temperature.

5.1.3.1 Bath heating proceeds at a rate consistent with the capabilities of the hot plate and prevents excessive loss of exothermic sample heat.

5.1.4 The high-temperature limit switch opens the supplementary circuit when the maximum safe bath temperature is reached.

5.1.5 Both the sample and bath have controlling thermocouples fed into the supplementary circuit and recording thermocouples fed into the multichannel recorder. Chromel-Alumel 20 gage, Type K thermocouples are suggested for this purpose.

5.2 *Suitable Circuit Diagram*, as provided in Fig. 2.

6. Materials

6.1 The heat-transfer medium shall be any suitable oil with a flash point well in excess of the maximum operating temperature of 250°C. The flash point of the oil should be checked periodically as it may decrease with use.

6.2 Borosilicate wool filtering fiber is used as the outer packing in the sample holder.

6.3 An inert fiber is used as the inner packing in the sample holder as a liquid adsorbant or powder dispersant.

6.4 A 13 mesh iron or nickel cylinder 2½ in. (63 mm) in

[2] A Type 2200 Thermolyne hot plate, available from VWR Scientific Inc., P.O. Box 3200, Rincon Annex, San Francisco, Calif. 94119, or equivalent, has been found suitable for this purpose.