Designation: D6801 – 07 (Reapproved 2015)

Standard Test Method for Measuring Maximum Spontaneous Heating Temperature of Art and Other Materials[1]

This standard is issued under the fixed designation D6801; 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 oil-based materials by exposure to elevated temperatures in air in a controlled semi-adiabatic system.

1.2 This test method has been developed to address an urgent need to identify oil-based materials that may require labeling for spontaneous heating tendency. Studies based on this test method may allow the development of a practice to identify such oil-based materials.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

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.* For safety concerns specific to disposal of solvent-soaked rags, see Appendix X1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E633 Guide for Use of Thermocouples in Creep and Stress-Rupture Testing to 1800°F (1000°C) in Air

E771 Test Method for Spontaneous Heating Tendency of Materials (Withdrawn 2001)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *maximum spontaneous heating temperature, n*—maximum temperature reached during spontaneous heating under the specified test conditions.

3.1.2 *oil-based materials, n*—seed and nut oils or materials that contain such oils.

3.1.3 *spontaneous heating, n*—exothermic reaction of a material due to slow or incomplete reaction that results in a temperature rise above that of its surroundings (see Test Method E771); also called *self heating*.

3.2 *Symbols:*

3.2.1 T_D—difference between maximum spontaneous heating temperature and control temperature.

3.2.2 T_S—maximum spontaneous heating temperature.

3.2.3 T_C—control temperature or maximum spontaneous temperature measured over 1 h immediately preceding the beginning of an experiment.

4. Summary of Test Method

4.1 A non-woven paper cloth is covered uniformly in 9.0 mL of test material mixed with 0.25 mL of 6 to 10 % manganese drier. This covered pad is then put in a small stainless steel holder with air holes in the sides. This holder is placed in a larger chamber, which is opened to the air from the top and heated until peak temperature is reached. The maximum spontaneous heating temperature inside the soaked paper cloth is recorded with a K thermocouple and maximum temperature recorder with the base temperature adjusted to 70 ± 2°C.

5. Significance and Use

5.1 This test method provides a means of accelerating the tendency of a material toward spontaneous heating that may eventually lead to a fire. It is applicable to liquids and pastes.

5.2 The spontaneous heating behavior of an oil-based material is affected by such factors as the availability of oxygen, the amount of driers present, the degree of polymerization of oils, the surface area of the cellulose material, measures to prevent heat dissipation, and the amount of oil in contact with cellulose material. The degree of spontaneous heating bears little relationship to the type of cellulose material to which an oil-based material comes in contact or whether or not oil

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.57 on Artist Paints and Related Materials.

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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.

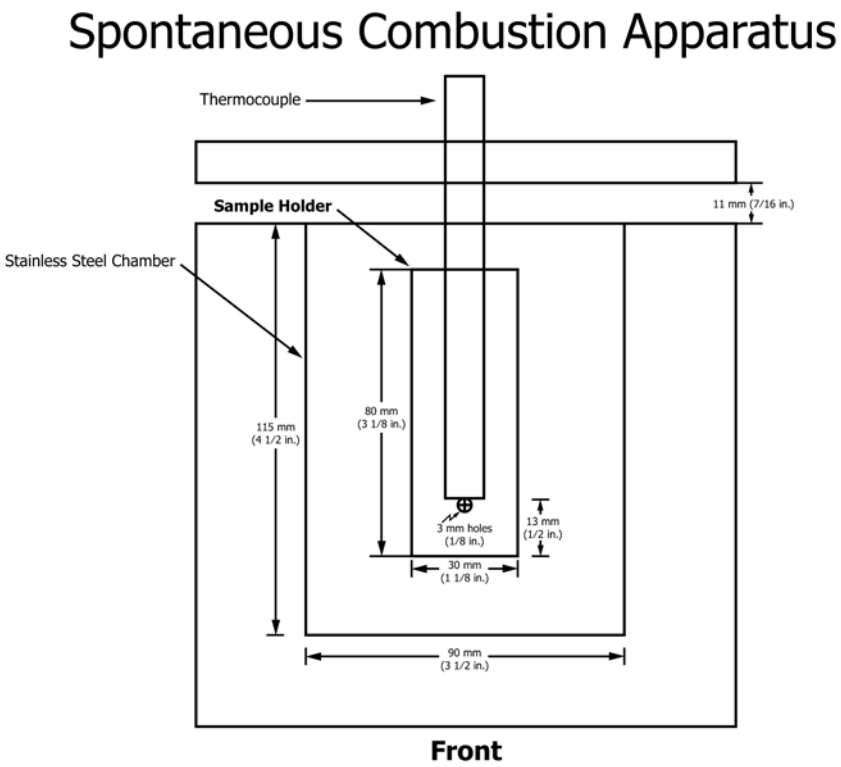

FIG. 1 Spontaneous Combustion Apparatus

soaked materials are first air dried. Small amounts of contaminants, such as oil paint, quartz dust, dirt or drier (for example, materials that might be found on a dirty, oil-soaked rag), can act as catalysts for this reaction.[4]

6. Apparatus

6.1 *Constant Temperature Water Bath*—set so that the base internal temperature of the test materials is 70 ± 2°C, see Fig. 1.

6.2 *K Thermocouple,* as described in Guide E633.

6.3 *Maximum Temperature Recorder,* which records maximum spontaneous heating temperature during a specified interval and is accurate to at least ±0.3°C and calibrated against NIST traceable standards.

6.4 *Stainless Steel Chamber for Water Bath*—See Fig. 1 for dimensions.

6.5 *Stainless Steel Holder (for paper cloth)*—Ten equally spaced 3-mm (⅛-in.) holes are drilled at 13 mm (½ in.) above the base. The holder is made with 2 mm (1⁄16 in.) stainless steel sheeting to form an open-topped box having a size of 70 mm (2¾ in.) wide by 80 mm (3⅛ in.) high by 30 mm (1⅛ in.) thick (see Fig. 1). Close seams with spot welding.

7. Materials

7.1 *Non-woven Paper Cloth* in rolls, 6.5 to 7.5 g/sheet. Sheet size is 20 by 30 cm (8 by 12 in.) and sheet density is 0.12 gm/cm^3.

7.2 *Manganese Drier* (manganese naphthenate-2-ethylhexanoate, 6 % Mn by weight) or manganese octanoate, 10 % Mn by weight.

8. Procedure

8.1 Fill water bath with water and adjust the heat until the center of a dry test cloth is at 70 ± 2°C.

8.2 Mix the liquid or paste material thoroughly with 0.25 mL the test material's weight of 6 to 10 % manganese drier.

8.3 Pipette liquids or use a spatula to spread pastes evenly over the surface of the non-woven paper cloth folded in ¼ ths and fold the soaked cloth in 1⁄16 ths.

8.4 Put the folded, soaked cloth in the stainless steel holder with the thermocouple placed centrally within the folded cloth (see Fig. 1).

8.5 After placing the cloth in the water bath chamber, partially open the lid (11 mm (7⁄16 in.)) to allow some airflow without loss of heat.

8.6 Record maximum spontaneous heating temperature with a K thermocouple and maximum temperature recorder until peak temperature is reached (usually 1.5 to 2 hours).

8.7 Record whether or not smoke is given off 15 min after peak temperature is reached and the degree blackening noted when the test cloth is unfolded with the following scale:

0	no charring
+	minimum charring
++	extensive charring
+++	extensive charring plus smoke

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1115. Contact ASTM Customer Service at service@astm.org.

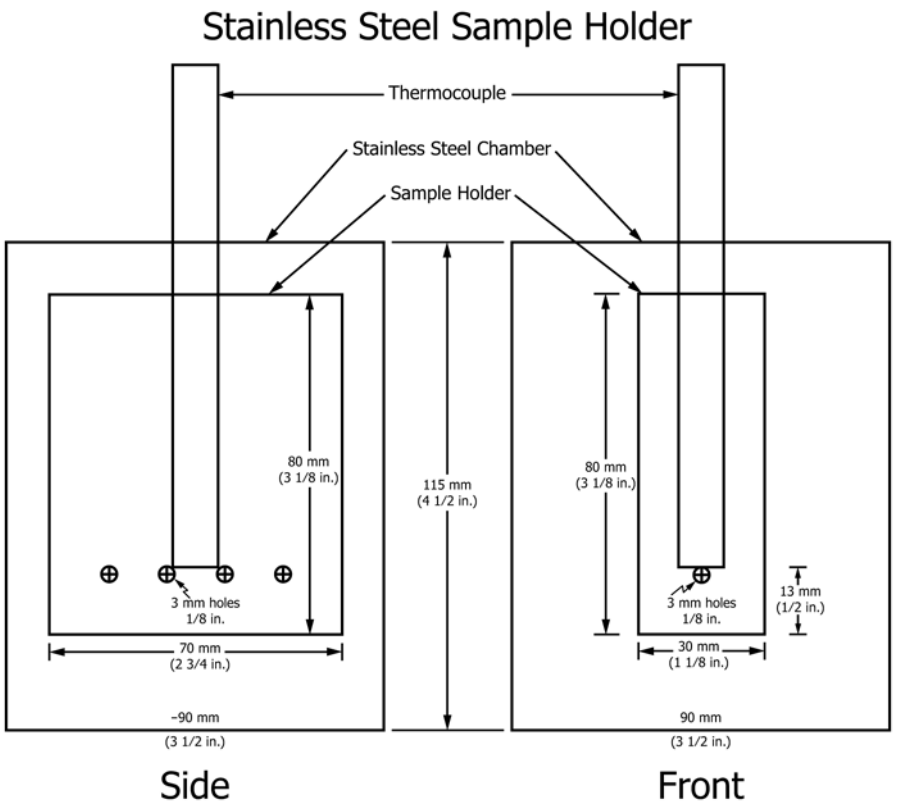

FIG. 1 Spontaneous Combustion Apparatus *(continued)*

8.8 **Warning**—This apparatus should not be left unattended during the course of a test.

9. Calculation and Interpretation

9.1 Calculate the temperature difference (T_D) between the maximum spontaneous heating temperature (T_S) and control temperature, T_C, as follows:

$$T_D = T_S - T_C \quad (1)$$

9.2 The greater the temperature difference (T_D), the greater the risk of spontaneous combustion associated with spontaneous heating.

10. Report

10.1 Report the following information:

10.1.1 Type of oil or material tested,

10.1.2 Maximum spontaneous heating temperature, and

10.1.3 Temperature difference (T_D) between control and test runs.

10.1.4 Degree of combustion.

11. Precision and Bias[4]

11.1 *Precision:*

11.1.1 *Repeatability*—The within laboratory standard deviation and 95 % repeatability limits for testing a liquid material are reported in Table 1.

TABLE 1 Within Laboratory Variability in Determination of Maximum Spontaneous Heating Temperature Using a Refined Linseed Oil/Manganese Drier Mix with Non-Woven Paper Cloth (8 df)[4,5]

	Control (°C) T_C	Maximum (°C) T_S	Difference (°C) T_D
Mean value	70.0	247	177
Repeatability standard deviation	1.4	6	6
Repeatability 95 % confidence limits	70.0 ± 3.9	247 ± 12	177 ± 12

11.1.2 *Reproducibility*—The between-laboratory standard deviations and 95 % reproducibility limits for testing three vegetable-based mediums and a paint are reported in Table 2.[5]

11.1.3 *Bias*—A low voltage to the maximum temperature recorder results in erroneously high maximum spontaneous heating temperature readings. To prevent bias, ensure that the battery meets manufacturer's specifications for voltage before each run or use a constant voltage source. Between laboratory variability increases for non-homogenous materials (alkyd medium, Table 2). Non-homogenous samples must be homogenized prior to testing.

12. Keywords

12.1 art materials; autocombustion; autoignition; flammability; ignition; spontaneous heating; vegetable oils

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1138. Contact ASTM Customer Service at service@astm.org.

TABLE 2 Between Laboratory Variability in Determination of Maximum Spontaneous Heating Temperature Difference (T_D)[4]

	Linseed oil #1	Linseed oil #2	Alkyd Medium	Alkyd Paint
Mean value (°C)	132.9	137.7	8.5	19.4
Degrees of freedom	2	2	2	2
Repeatability standard deviation	4.2	3.6	2.2	5.4
Repeatability 95 % limits	±8.4	±7.2	±4.4	±10.8
Reproducibility standard deviation	11.3	16.1	8.8	6.3
Reproducibility 95 % limits	±22.6	±32.2	±17.6	±12.3

APPENDIX

(Nonmandatory Information)

X1. DISPOSAL OF OIL OR SOLVENT-SOAKED RAGS

X1.1 For oils materials that present a risk of spontaneous combustion, special disposal procedures are necessary to decrease risk. Hanging contaminated rags out to "dry" is ineffective at decreasing risk.[4] In order to prevent unexpected fires, store water-damped contaminated rags in a metal container with an air-tight top. Alternately, washing such rags will remove contaminating materials and eliminate risk.