Designation: D 6801 – 02a

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

This standard is issued under the fixed designation D 6801; 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 art 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 art materials that may require labeling for spontaneous heating tendency. Studies based on this method may allow the development of a practice to identify such oil-based art materials.

1.3 The values stated in SI units are to be regarded as the standard. The inch-pound 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 the appendix.

2. Referenced Documents

2.1 *ASTM Standards:*

E 633 Guide for Use of Thermocouples in Creep and Stress Rupture Testing to 1000°C (1800°F) in Air[2]

E 771 Test Method for Spontaneous Heating Tendency of Materials[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 art materials, n*—vegetable oils that are used as art materials or art materials that contain vegetable 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 E 771); 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 1.2 to 1.5 times its weight in a oil-based art material mixed with 6–10 % manganese drier at a 1:45 to 1:50 ratio, drier to art material. 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 for 7 to 15 h in a 70°C water bath. The maximum spontaneous heating temperature inside the soaked paper cloth is recorded with a K thermocouple and maximum temperature recorder.

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 art 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 rate and magnitude of spontaneous heating bears little relationship to the type of

[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.

Current edition approved Dec. 10, 2002. Published February 2003. Originally approved in 2000 as PS 124 - 00. Last previous edition approved in 2002 as D 6801 - 02.

[2] *Annual Book of ASTM Standards*, Vol 03.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

cellulose material to which an oil-based art material comes in contact or whether or not oil soaked materials are first air dried.[4]

6. Apparatus

6.1 *Constant Temperature Water Bath*—water bath that can maintain water temperature at 70 ± 2°C, see Fig. 1.

6.2 *K Thermocouple*, as described in Guide E 633.

6.3 *Maximum Temperature Recorder*, which records maximum spontaneous heating temperature during a specified interval.

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.) aluminum 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 aluminum 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.

[4] Supporting data are available from ASTM International Headquarters. Request RR: D01–1115.

8. Procedure

8.1 Fill water bath with water and heat until water is at 70°C ± 2°C.

8.2 Weigh the non-woven paper cloth. Prepare 1.2 to 1.5 times its weight of liquid or paste art material.

8.3 Mix the liquid or paste art material thoroughly with 6–10 % manganese drier in a ratio of 1:45 to 1:50 drier to art material.

8.4 Pipette liquids or use a spatula to spread pastes evenly over the non-woven paper cloth and fold the soaked cloth in 8ths so that it will fit into the stainless steel holder.

8.5 Put the folded, soaked cloth in the aluminum holder with the thermocouple placed centrally within the folded cloth (see Fig. 1).

8.6 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.

8.7 Record maximum spontaneous heating temperature with a K thermocouple and maximum temperature recorder during the first 75 min and during the subsequent 7 to 15 h.

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

9. Calculation or Interpretation of Results

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

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

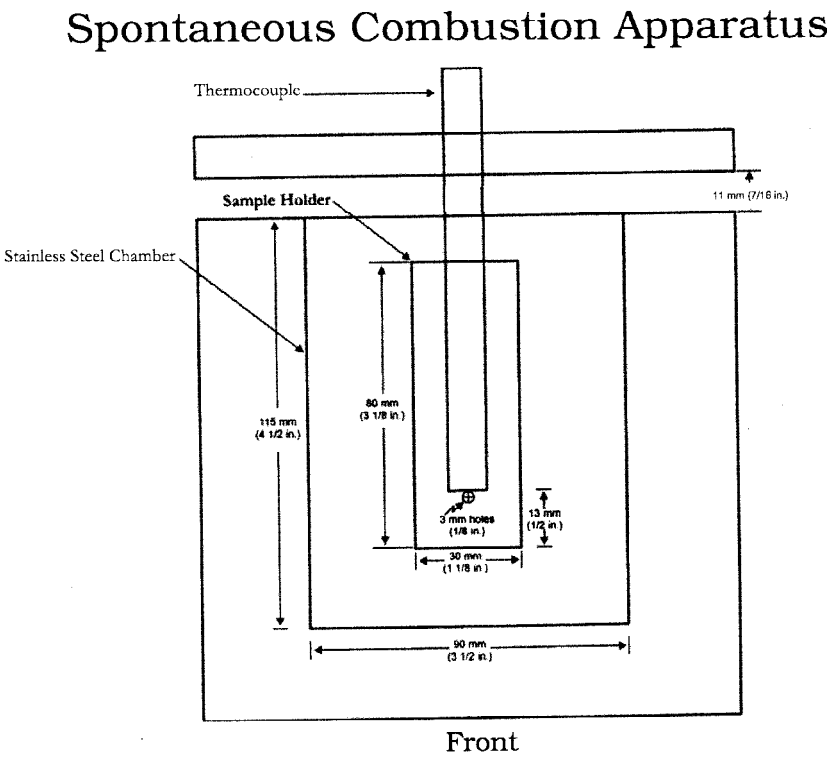

FIG. 1 Spontaneous Combustion Apparatus

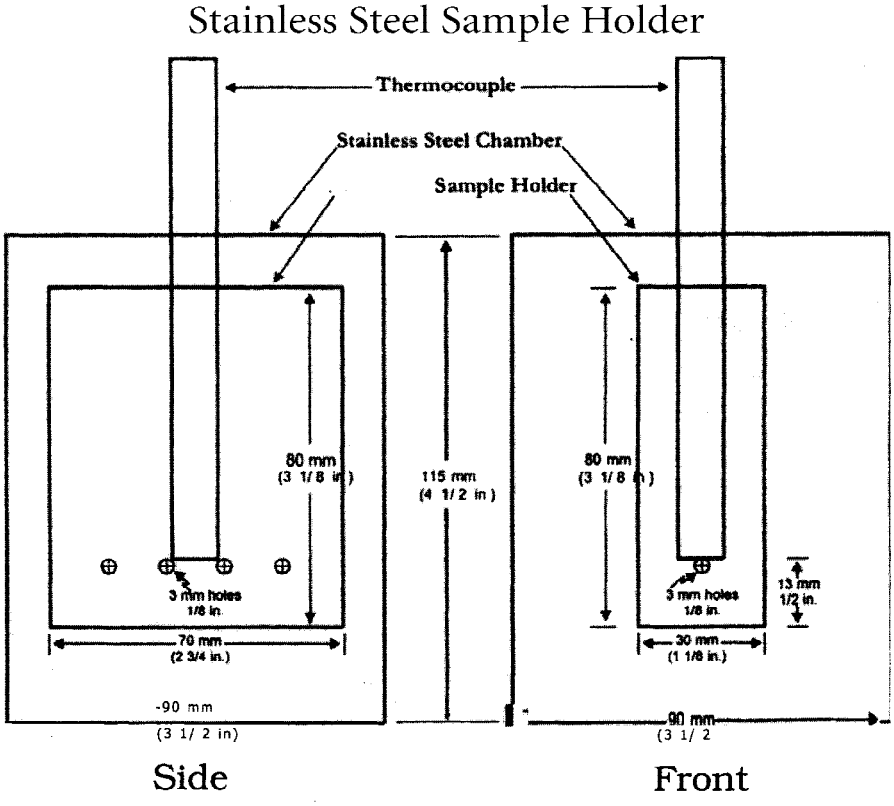

FIG. 1 *(continued)*

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 art material tested,

10.1.2 Maximum spontaneous heating temperature, and

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

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 art 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)

	Control (°C) T_C	Maximum (°C) T_S	Difference (°C) T_D
Mean value	67.2	196	129
Repeatability standard deviation	1.4	25	25
Repeatability 95 % confidence limits	±3.9	±70	±70

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

11.1.3 *Bias*—A low voltage to the thermocouple thermometer 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

TABLE 2 Between Laboratory Variability in Determination of Maximum Spontaneous Heating Temperature Difference (T_I)[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 and art 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 contaminated rags in a water-filled metal container with an air-tight top. Alternately, washing such rags will remove contaminating materials and eliminate risk.