Designation: D 3850 – 94 (Reapproved 2000)

An American National Standard

Standard Test Method for Rapid Thermal Degradation of Solid Electrical Insulating Materials By Thermogravimetric Method (TGA)[1]

This standard is issued under the fixed designation D 3850; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method outlines a procedure for obtaining thermogravimetric (TGA) data on solid polymeric materials intended for use as electrical insulating materials.

1.2 Do not use this standard to quantify an estimate of the long-term thermal capability for any electrical insulating material. If a relationship exists between TGA and the long-term thermal capabilities of a material, then that fact must be established and made public, preferably by comparing data between a candidate and another material known to display similar failure modes.

1.3 The values stated in SI units are the standard.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics[2]

D 1600 Terminology of Abbreviated Terms Relating to Plastics[2]

D 1711 Terminology Relating to Electrical Insulation[3]

D 2307 Test Method for Relative Thermal Endurance of Film-Insulated Round Magnet Wire[3]

E 220 Method for Calibration of Thermocouples by Comparison Techniques[4]

E 473 Terminology Relating to Thermal Analysis[5]

E 914 Practice for Evaluating Temperature Scale for Thermogravimetry[5]

E 1582 Practice For Calibration of Temperature Scale For Thermogravimetry[5]

3. Terminology

3.1 *Definitions*—Definitions are in accordance with Terminology D 883, Terminology D 1711, and Terminology E 473.

3.2 *Abbreviations*—Abbreviations are in accordance with Terminology D 1600, unless otherwise indicated.

4. Summary of Test Method

4.1 This thermogravimetric technique uses the record of the mass loss versus the temperature of the specimen during the time of exposure to a specified prescribed environment using a controlled time rate of heating.

4.2 The record is a TGA curve, with percent of initial mass as the ordinate and temperature as the abscissa (see Figs. 1 and 2).

4.3 The temperature is measured and recorded at specified mass loss points (recorded as a TGA curve), using an electronic chart recorder or other suitable data acquisition device.

5. Significance and Use

5.1 Thermogravimetry is useful in determining the dynamic functional effect of temperature on the amount of volatile materials leaving a specimen as the latter is heated progressively to higher temperatures. TGA can be useful for process control, process development, material evaluation, and for identification and quality control in specifications.

5.2 The thermal stability of a material can be associated with the degree and time rate of mass loss as a function of temperature. TGA curves can, therefore, be used as a preliminary screen method in the evaluation of relative behavior of insulating materials of the same generic family.

5.3 The functional temperature-life relationship of an insulating material in any given application depends on a number of service and environmental factors. Therefore, the information obtained from TGA curves is not adequate by itself to describe the thermal capability of an insulating material.

5.4 Refer to the Appendix for further discussion of the interpretation of TGA data.

6. Apparatus

6.1 *Thermogravimetric Analyzer*—A system of related instruments comprising:

6.1.1 *Microbalance*, of the null type, sensitive to 0.001 mg,

[1] This test method is under the jurisdiction of ASTM Committee D09 on Electrical and Electronic Insulating Materials and is the direct responsibility of Subcommittee D09.17 on Thermal Capabilities.

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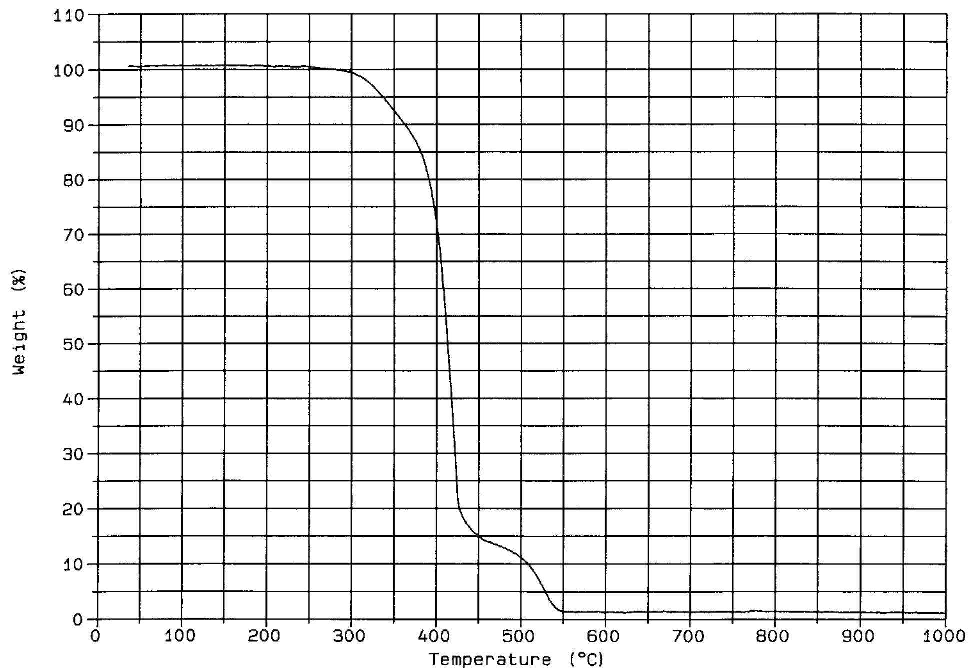
[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 10.01.

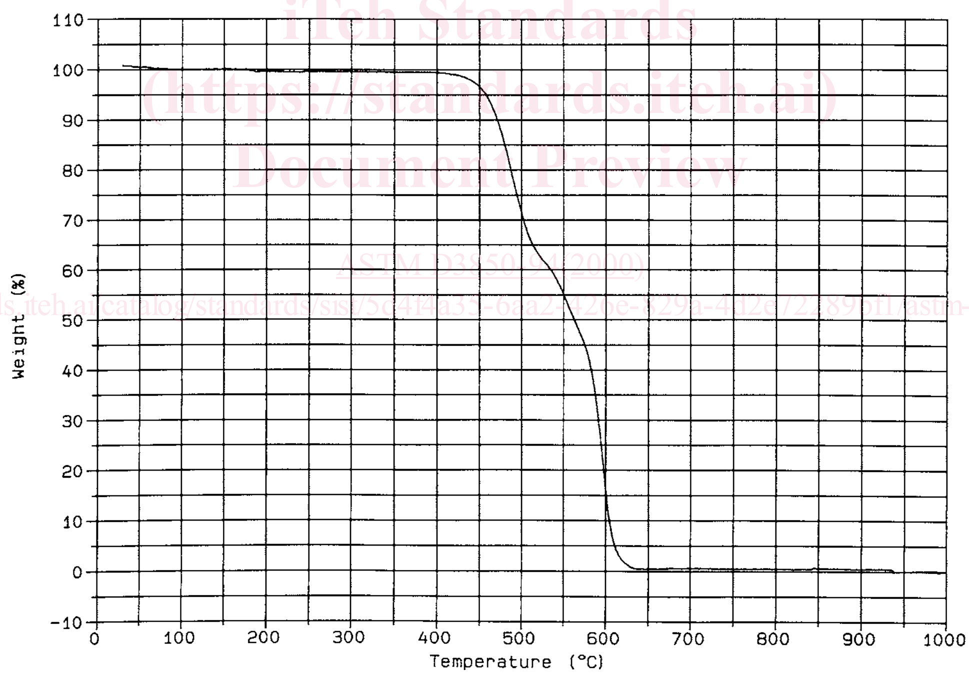
[4] *Annual Book of ASTM Standards*, Vol 14.03.

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



Sample 8.54 mg Heating Rate 5°C/min Purging Gas Flow 0.8 mL/s

FIG. 1 Curve No. 1, Typical TGA for Polyester Film



Sample 5.93 mg Heating Rate 5°C/min Purging Gas Flow 0.8 mL/s

FIG. 2 Curve No. 2, Typical TGA for Polyimide Film

6.1.2 *Furnace*, controllable at a constant rate over a temperature range of interest, typically 25 to 1000°C,

6.1.3 *Temperature Programmer*, capable of providing a linear rate of rise of the furnace at a predetermined value (normally 5°C/min) with a tolerance of ± 0.1°C/min,

6.1.4 *Suitable Data Acquisition Device*, and

6.1.5 *Supply of Purging Gas*.

NOTE 1—For many applications, the purging gas is nitrogen or air having a dew point of at or below −10°C.

7. Sampling

7.1 Use sampling plans as described in specifications or test methods specific to individual electrical insulating materials.

8. Test Specimens

8.1 Prepare test specimens in accordance with the test method applicable to the material under investigation.