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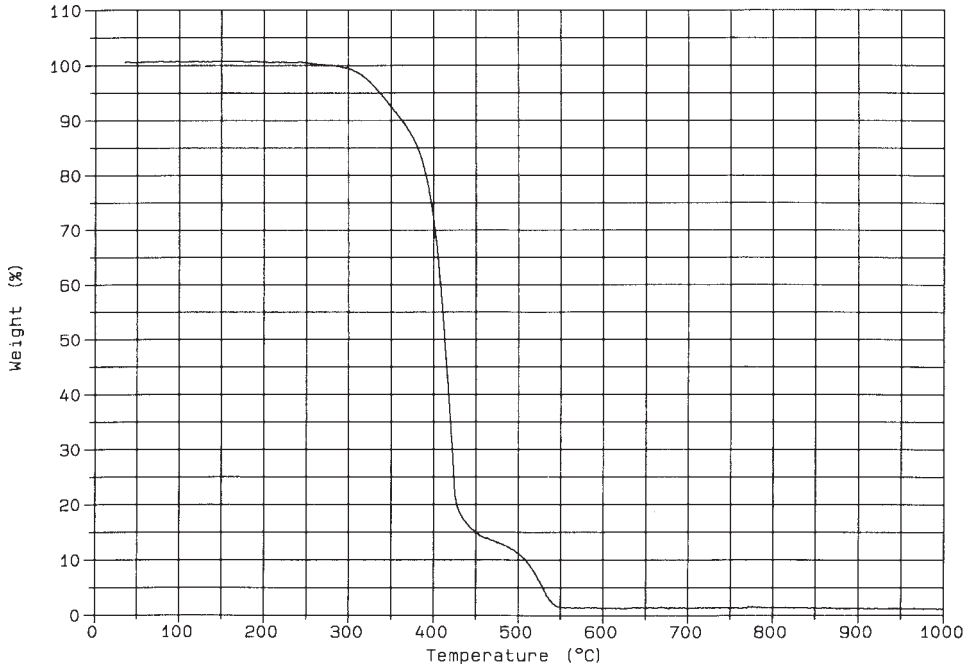
[2] *Annual Book of ASTM Standards*, Vol 08.01.

[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.

8.2 Generally, it is found that specimens of 2 to 20 mg are satisfactory, depending on the configuration and test apparatus. Test results depend in part on the size and shape of specimen, due to thermal equilibrium and diffusion effects.

8.3 When the specimen is a coating on a substrate, the total mass may be substantially greater, because of the mass contribution of the substrate material.

9. Procedure

9.1 Calibrate the balance at full scale to within ± 0.01 mg, following the recommended procedure.

9.2 Calibrate the temperature-sensing system to within ± 1°C (see Method E 220), following the recommended procedure.

9.2.1 Position the temperature sensor to prevent contact with specimens which may become distorted during heating.

9.2.2 Temperature calibration is critical and the method employed will vary with the apparatus. Calibrate in accordance with Practice E 1582.

9.3 Adjust the purge rate to the specified value.

9.4 Adjust the *Y* axis (mass) to chart zero.

9.5 Adjust the *X* axis to the required temperature range.

9.6 Place the specimen in the specimen holder and record the initial mass.

9.7 Set the heating rate to 5°C/min rate of rise.

9.8 Start the heating program and record the mass change until there is no further mass loss.

NOTE 2—Normally the purge rate is 0.7 to 1.6 mL/s.

10. Report

10.1 Report the following information:

10.1.1 Identification of the sample and apparatus,

10.1.2 Curing time and temperature in the case of resin specimens,

10.1.3 Mass, approximate dimensions and form (for example, film, laminate, molded) of the specimen,

10.1.4 Heating rate,

10.1.5 Rate of flow and type of gas used for purging,

10.1.6 TGA curve of material evaluated, and

10.1.7 Temperatures at which losses of initial specimen mass, if obtained, of 10, 20, 30, 50, and 75 % occur.

NOTE 3—Do not list temperatures that exceed the resolution of the instrumentation. Normally this is not to be greater than 2.5°C. Report the resolution.

11. Precision and Bias

11.1 This test method is based on the dynamic measurement of mass loss as a function of increasing temperature. Deviations in results that affect precision are caused by variations in a number of complex factors (for example, physical irregularities of the specimen, variations in the purging gas composition and flow characteristics) and generally will not correlate simply with changes in these factors.

11.2 The repeatability of the mass loss measurements as a function of temperature within one laboratory (and one apparatus) is approximately ± 5°C.

11.3 Limited inter-laboratory testing done as a preliminary preparation for this test method indicate that mass loss measurements plotted as a function of temperature have a reproducibility of ± 25°C.

11.4 This test method has no bias because the thermal degradation characteristic is defined by the method.

12. Keywords

12.1 degradation; insulating; mass loss; polymeric material; thermogravimetric analysis; TGA

APPENDIX

(Nonmandatory Information)

X1. INTERPRETATION OF THERMOGRAVIMETRIC TEST TECHNIQUE

X1.1 *Introduction*—Thermogravimetry is the continuous measurement of mass loss of a specimen as the temperature is increased at a specific rate. Since the test method requires the continuous measurement of a varying mass and temperature and the control of temperature rate of rise, differences may exist between instruments, experimenters, or both, even when the precision of the individual component sensors are known. Calibration of the instrumentation system is based upon a comparison under dynamic conditions.

X1.2 *Calibration*—Initial calibration should follow the recommended procedure (when available). This procedure should ensure that individual sensors are correct. The relationship between the temperature sensor, usually a thermocouple, and the specimen design and the type of atmosphere, including the rate of flow of the gas through the weighing chamber, will affect the overall calibration of the system.

X1.3 *Atmosphere*—The rate of mass loss is dependent in part upon the atmosphere to which the test specimen is exposed. For the best correlation of test results to end use, purge with the air or other gas that relates to the end use conditions. The rate of flow of the gas in the cell will have a significant effect on the calibration of the system. It is, therefore, necessary to select the rate of flow, usually 0.7 to 1.6 mL/s, prior to calibration of the system. After calibrating the system, do not change the flow rate.

X1.4 *Specimen Design*, is dictated by the material under consideration, material application, and the instrumentation. Use a specimen in the form normally found in use (for example, film and coatings). The size will depend on the instrumentation to some degree. The surface area will affect the overall results. For instance, if a specimen with a large surface is compared to one with smaller surface area, both of the same

mass, the small surface area specimen will normally lose mass at a slower rate, due to thermal equilibrium and thermal effects. Select the specimen configuration and mass prior to system calibration.

X1.5 *System Calibration:*

X1.5.1 System calibration of the TGA instrumentation incorporates the comparison of specimen temperature to a measured physical change in the specimen. The weakest point in the calibration procedure is the temperature sensor, usually a thermocouple. Thermocouples are nonlinear within the normal range of operating temperatures, and after a few times of operation may drift from calibration. The location of the temperature sensor in the weighing chamber must be such that it will provide the best estimate of the specimen temperature. Since no standard measurement will provide this location, it is necessary to make a comparison test.

X1.5.2 Practice E 1582 can be used for temperature calibration.

X1.5.3 Differences between laboratories may always exist. Comparison to the referenced curves or another mutually selected TGA curve will provide a reasonably accurate method for communicating various TGA data. Close attention to the overall calibration on a continual basis will ensure good repeatability within one laboratory over a long period of time.

X1.6 *Interpretation of TGA Data*—Thermogravimetry is a relatively fast means of comparing materials. The dynamic relationship between mass loss and temperature is the only thermal characteristic considered with TGA. How this temperature-mass loss characteristic affects the use of an electrical insulation in the application is unknown. The rate of mass loss affects electrical insulation life. Mass loss that will result in failure varies with material and temperature. In addition, the mass loss required for a failure varies with the failure mode. Due to the many unknown factors in any given application, the exact relationship between mass loss and failure mode for each application should be determined experimentally. Generally, material comparisons by TGA have not always been in the same order as the known temperature class. As an example, the round robin testing performed during the development of this test method indicated that polyamide film was thermally superior to a PET film. Both life tests and experience have verified that PET film is thermally superior to polyamide film in a variety of electrical applications.

X1.6.1 In 1967, Sweitzer and Stugart[6] illustrated that an equivalent polyamide polymer for magnet wire yielded higher TGA data and had a lower thermal life (Test Method D 2307) in comparison to the same polyamide cured under different conditions.

X1.6.2 Thermogravimetry is an important tool in observing the effect of polymer variation due to changes in one thermal characteristic. The relationship between thermogravimetry and application life is unknown.

NOTE X1.1—The attached Figs. 1 and 2 for polyester film and polyimide film are illustrative of the original round-robin test work carried out in the preparation of this test method.

[6] Sweitzer and Stugart, "Screening Polymers for Use as Magnet Wire Enamel," *Proceeding of the Seventh Electrical Insulation Conference*, 1967, Paper No. IEEE 32C-79-64.