Designation: D 2584 – 02

An American National Standard

Standard Test Method for Ignition Loss of Cured Reinforced Resins[1]

This standard is issued under the fixed designation D 2584; 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 covers the determination of the ignition loss of cured reinforced resins. This ignition loss can be considered to be the resin content within the limitations of 4.2.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics for Testing[2]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

3. Summary of Test Method

3.1 The specimen contained in a crucible is ignited and allowed to burn until only ash and carbon remain. The carbonaceous residue is reduced to an ash by heating in a muffle furnace at 565°C (1050°F), cooled in a desiccator, and weighed.

4. Significance and Use

4.1 This test method can be used to obtain the ignition loss of a cured reinforced resin sample.

4.2 If only glass fabric or filament is used as the reinforcement of an organic resin that is completely decomposed to volatile materials under the conditions of this test and the small amount of volatiles (water, residual solvent) that may be present is ignored, the ignition loss can be considered to be the resin content of the sample.

4.2.1 This test method does not provide a measure of resin content for samples containing reinforcing materials that lose weight under the conditions of the test or containing resins that do not decompose to volatile materials released by ignition.

5. Apparatus

5.1 *Crucible*, platinum or porcelain, approximately 30-mL capacity.

5.2 *Electric Muffle Furnace*, capable of maintaining a temperature of 565 ± 28°C (1050± 50°F).

6. Test Specimen

6.1 A minimum of three specimens shall be tested for each sample.

NOTE 1—It is often convenient to use samples obtained from specimens that have been tested for mechanical properties such as flexural or tensile strength. Specimens obtained from these samples must be dry and the fractured areas removed, leaving square, unfrayed faces, before being weighed and ignited.

6.2 The specimen shall weigh approximately 5 g with a maximum size of 2.5 by 2.5 cm by thickness (1 by 1 in. by thickness).

NOTE 2—Materials that have gross differences in the ratio of resin to reinforcement within an area as small as 2.5 by 2.5 cm (1 by 1 in.) may require a larger specimen area than that listed in 6.2. If larger specimens are utilized, it will be necessary to cut into approximately 2.5 by 2.5-cm (1 by 1-in.) pieces and place in a crucible of sufficient size to contain the specimen.

7. Conditioning

7.1 *Conditioning*—Condition the test specimens at 23± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D 618 for those tests where conditioning is required. In cases of disagreement, the tolerances shall be ±1°C (±1.8°F) and ±2 % relative humidity.

7.2 *Test Conditions*—Conduct tests in the standard laboratory atmosphere of 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity, unless otherwise specified in the test methods or in the specification. In cases of disagreement, the tolerances shall be ±1°C (±1.8°F) and ±2 % relative humidity.

8. Procedure

8.1 Heat a crucible at 500 to 600°C for 10 min or more.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.18 on Reinforced Thermosetting Plastics.

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

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

***A Summary of Changes section appears at the end of this standard.**

Cool to room temperature in a desiccator and weigh to the nearest 1.0 mg.

8.2 Place the specimen in the crucible and weigh to the nearest 1.0 mg. Heat the crucible and specimen in a bunsen flame until the contents ignite. Maintain such a temperature that the specimen burns at a uniform and moderate rate until only ash and carbon remain when the burning ceases.

NOTE 3—It is not absolutely necessary to ignite the specimen in a bunsen flame. Instead the crucible and contents can be placed in a muffle furnace at a temperature lower than 565°C and ignited. Care must be taken that the ignition does not proceed so rapidly that there will be a mechanical loss of the noncombustible residue.

8.3 Heat the crucible and residue in the muffle furnace at 565 ± 28°C (1050 ± 50°F) until all carbonaceous material has disappeared (Note 4). Cool the crucible to room temperature in a desiccator and weight to the nearest 1.0 mg.

NOTE 4—The time for the carbonaceous residue to disappear is dependent largely on the specimen geometry. It can be up to 6 h but is usually much less.

8.4 Bring the crucible and residue to constant weight within 1.0 mg.

9. Calculations

9.1 Calculate the ignition loss of the specimen in weight percent as follows:

$$\text{Ignition loss, weight \%} = [(W_1 - W_2)/W_1] \times 100 \quad (1)$$

where:
W_1 = weight of specimen, g, and
W_2 = weight of residue, g.

9.2 Average the 3 specimen values to obtain the sample average.

$$s = \sqrt{[\Sigma X^2 - n(\bar{X})^2]/(n-1)} \quad (2)$$

where:
s = estimated standard deviation,
X = value of a single observation,
n = number of observations,
$\bar{X}$ = arithmetic mean of the set of observations.

9.3 Subtract the lowest specimen ignition loss from the highest specimen ignition loss for the sample and report as the ignition loss range.

10. Report

10.1 Report the following information:

10.1.1 Complete identification of the material.

10.1.2 The ignition loss, weight percent of the sample, and standard deviation. If only glass reinforcement and organic resin were present, the ignition loss can be considered to be the resin content.

10.1.3 Observations in regard to any irregularities noted in the physical properties of the residue, such as melting.

11. Precision and Bias

11.1 Table 1 is based on a round robin conducted in 2001 in accordance with Practice E 691, involving two materials tested by seven laboratories (six for pultruded rod). For each material, all the samples were prepared at one source, but the individual specimens were prepared at the laboratories which tested them. Each test result was the average of three individual determinations. Each laboratory obtained two test results for each material.

TABLE 1 Ignition Loss of Cured Reinforced Resin

Material	Values expressed in % loss				
	Average	S_r[A]	S_r[B]	R[C]	R[D]
Glass Reinforced Laminate	36.30	2.57	2.57	7.21	7.21
Pultruded Rod	21.36	0.289	0.940	0.0808	0.2632

[A] S_r = within laboratory standard deviation for the indicated material. It is obtained by pooling the within-laboratory standard deviations of the test results for all of the participating laboratories:

$$S_r = [[(S_1)^2 + (S_2)^2 .... + (S_n)^2]/n]^{\frac{1}{2}}$$

[B] S_R = between-laboratories reproducibility, expressed as standard deviation:

$$S_R = [S_r^2 + S_L^2]^{\frac{1}{2}}$$

where S_L = standard deviation of laboratory means.

[C] r = within-laboratory critical interval between tow test results = 2.8 × S_r.

[D] R = between-laboratories critical interval between tow test results = 2.8 × S_R.

NOTE 5—**Caution:** The explanation of "r" and "R" in 11.2 and 11.3 are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 should not be applied to acceptance or rejection of materials, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots, formulations, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their materials and laboratory (or between specific laboratories). The principles of 11.2-11.2.3 would then be valid for such data.

11.2 *Concept of "r" and "R" in Table 1*—If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from testing two specimens for each test result, then:

11.2.1 *Repeatability*:

Two results obtained within one laboratory shall be judged not equivalent if they differ by more than the "r" value for that material. "r" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

11.2.2 *Reproducibility*:

Two test results obtained by different laboratories shall be judged not equivalent if they differ by more than the "R" value for that material. "R" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

11.2.3 Any judgement in accordance with 11.2.1 or 11.2.2 would have an approximate 95% (0.95) probability of being correct.

11.3 There are no recognized standards by which to estimate bias of this method.

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

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(*1*) A new precision and bias statement was prepared following a round robin in 2001.