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Standard Test Methods for
Void Content of Reinforced Plastics[1]

This standard is issued under the fixed designation D 2734; 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 These test methods cover the void content of reinforced plastics or "composites." The test methods are applicable to composites for which the effects of ignition on the materials are known. Most plastics, glass, and reinforcements fall into this class. These test methods are not applicable to composites for which the effects of ignition on the plastics, the reinforcement, and any fillers are unknown. This class may include silicone resins, which do not burn off completely, reinforcements consisting of metals, organic materials, or inorganic materials which may gain or lose weight, and fillers consisting of oxides, carbonates, etc., which may gain or lose weight. Note that separate weight loss tests of individual materials will usually, but not necessarily, give the same result as when all the materials are combined.

Note 1—There is no known ISO equivalent to these test methods.

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 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:*[2]
D 618 Practice for Conditioning Plastics for Testing
D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
D 1505 Test Method for Density of Plastics by the Density-Gradient Technique
D 2584 Test Method for Ignition Loss of Cured Reinforced Resins

3. Summary of Test Methods

3.1 The densities of the resin, the reinforcement, and the composites are measured separately. Then the resin content is measured and a theoretical composite density calculated. This is compared to the measured composite density. The difference in densities indicates the void content. A good composite may have 1 % voids or less, while a poorly made composite can have a much higher void content. Finite values under 1 % should be recognized as representing a laminate density quality, but true void content level must be established by complementary tests or background experience, or both.

4. Significance and Use

4.1 The void content of a composite may significantly affect some of its mechanical properties. Higher void contents usually mean lower fatigue resistance, greater susceptibility to water penetration and weathering, and increased variation or scatter in strength properties. The knowledge of void content is desirable for estimation of quality of composites.

5. Interferences

5.1 The density of the resin, in these test methods, is assumed to be the same in the composite as it is in a large cast mass. Although there is no realistic way to avoid this assumption, it is nevertheless not strictly correct. Differences in curing, heat and pressure, and molecular forces from the reinforcement surface all change the composite resin density from the bulk resin density. The usual change is that bulk density is lower, making void content seem lower than it really is.

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[1] These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.18 on Reinforced Thermosetting Plastics.
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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.

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

5.2 For composites with high void contents, this error will lower the true value an insignificant amount, from a true 7 % down to a calculated 6.7 %, for example. For composites with low and void contents, the value may be lowered from a true 0.2 % to a calculated − 0.1 %. This would indicate an obvious error, and illustrates that as the void content gets lower the constant error in resin density gets progressively more important. Note that these values are for example only, that different resin systems can give different errors, and that it is left to the individual tester to determine the accuracy of the calculated result in his particular measurement.

5.3 For the special case of semi-crystalline plastics, such as polyphenylene sulfide (PPS) and polyetheretherketone (PEEK), an interference due to the level of crystallinity present in the composite can cause significant variation in the measurement of void content by this test method. The level of crystallinity can be affected by a variety of circumstances, including the molding conditions. For these polymers, the density used in the calculation must be the actual density of the resin in the composite.

NOTE 2—The actual degree of crystallinity of the composite can be measured by techniques such as differential scanning calorimetry (DSC) or by X-ray diffraction.

6. Conditioning

6.1 *Conditioning*—Condition the test specimens at 23 ± 2°C [73.4(73.4 ± 3.6°F]3.6°F) and 50 ± 5%10 % 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](1.8°F) and ±2 % relative humidity.

6.2 *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. In cases of disagreement, the tolerances shall be 1°C (1.8°F) and ±2%±5 % relative humidity.

7. Procedure

7.1 *Density of the Resin and the Composite*—Three test methods are presented for these measurements. Measure the density on pieces of resin that are bubble-free and that were cured under heat, time, and pressure conditions that are as close as practicable to the conditions under which the composite was cured. Density measurements supplied by the resin manufacturer are acceptable if they are certified for each batch.

7.1.1 *Test Method A*—Measure densities using Test Methods D 792. Paragraph 1.1 of that test method requires the specimen to have smooth edges and surfaces. For composites, this requirement may necessitate hand sanding the specimen with 400-grit emery paper to remove fuzzy edges caused by cutting. The specimen should be as free as possible from geometric irregularities which tend to trap air bubbles. Remove any bubbles by a wire, or other mechanical means. Do not use a vacuum to remove bubbles, because cut surfaces of composites may be porous and exposure to a vacuum will force water into the pores, causing an error in the density measurement. In some cases of extreme porosity, just immersing the composite in water, without using a vacuum, will allow an unacceptably large amount of water takeup by the composite. In these cases, seal the porous surfaces by coating them with a known amount of sealer of known density. This will require weighing the specimen, then spraying-on or wiping-on the sealer, then reweighing. A correction for this added material will then be required in the calculations.

7.1.2 *Test Method B*—Measure densities in accordance with Test Method D 1505. The comments in 7.1.1 concerning fuzzy edges and removal of bubbles apply here also.

7.1.3 *Test Method C*:

7.1.3.1 Densities calculated from weight and volume measurements are acceptable if the specimens are smooth, uniform, and of such shape that the volume can be calculated accurately from the dimensions.

7.1.3.2 *Procedure*—The volume of each specimen shall not be less than 2 cm³ [0.125 in.(0.125 in.³]). Make dimensional measurements with a micrometer at all edges (12 in all for a 6-sided rectangular block). Use the averages for each dimension to calculate the volume.

7.1.3.3 The tolerance on the accuracy of the micrometer measurements shall be ±0.0013 cm [±0.0005 in.](±0.0005 in.). With maximum tolerance buildup on a small sample, this could result in an error in the calculated volume of 0.6 %. For larger samples, and with some measurements being in error on the plus side and some on the minus side, the error in the calculated volume should not exceed 0.2 %.

7.1.3.4 Calculate the density by dividing the weight by the volume; express as grams per cubic centimetre.

7.2 *Density of the Glass or Other Reinforcement*—Most glass reinforcement is E glass, which typically has a density between 2.54 and 2.59 g/cm³ ; S glass density is 2.46 to 2.49 g/cm³. However, if a density determination is necessary, use Test Methods D 792. Pay particular attention to Note 11 of that test method, which discusses removal of trapped air by exposure of the sample to a vacuum. This step can be assumed to be necessary in every determination of glass density. Use a vacuum of 3 mm Hg or better. Several cycles of atmospheric pressure-to-vacuum may be required before the trapped air is completely removed.

NOTE 3—It is suggested that the density of the glass supplied be verified with the glass fiber producer.

7.3 *Resin Content of Composite*—Determine in accordance with Test Method D 2584. The ignition loss in that test method is the resin content of the sample and is to be recorded as the weight percent as indicated.

8. Theoretical Density

8.1 *Calculation*—Using the values determined in 7.1, 7.2, and 7.3, calculate theoretical density of a composite as follows:

2