

Designation: D494 – 11

Standard Test Method for Acetone Extraction of Phenolic Molded or Laminated Products¹

This standard is issued under the fixed designation D494; 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 the determination of the amount of acetone-soluble matter in molded or laminated phenolic products.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered standard.

1.3 This standard does not purport to address all of the safety problems, 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.

NOTE 1-This test method is similar to ISO 308.

2. Referenced Documents

2.1 ASTM Standards:²

D618 Practice for Conditioning Plastics for Testing

D883 Terminology Relating to Plastics

E11 Specification for Woven Wire Test Sieve Cloth and Test
Sieves and ards, iteh ai/catalog/standards/sist/50b74107
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 ISO Standards:

ISO 308 Plastics—Phenolic moulding materials— Determination of acetone-soluble matter (apparent resin content of material in the unmoulded state)

3. Terminology

3.1 All definitions are in accordance with Terminology D883.

4. Significance and Use

4.1 For molded phenolic products, acetone extraction shall be considered solely as a quantitative expression of a property normally associated with degree of cure. There is no demonstrably rigorous relation between the optimum mechanical and electrical properties of a well-cured piece and the numerical value of the acetone test. The amount of acetone-soluble matter is affected by: (1) nature of resin and filler, (2) lubricant, (3) molding temperature, (4) length of cure, (5) thickness of the section from which sample is taken, (6) nature of molded piece, (7) technique used in molding, (8) distribution of fines in the material to be extracted, and (9) method of grinding the specimen. These variations under some conditions will cause a difference of 3 to 4 % in acetone-extractable matter. For this reason, the test method shall be used only as a comparative test for measuring undercure.

4.2 For laminated phenolic products, acetone extraction indicates change in stage of cure, change in resin content, change in type of resin used, presence of plasticizers or other acetone-extractable addition agents, and is affected in general by the same factors as stated in 6.1

8b87-4fea-81f2-74054e5ebbd4/astm-d494-11 5. Apparatus

5.1 Sieves—The set of sieves used shall consist of sieves Nos. 40 (425-µm) and 140 (106-µm), with a cover and receiving pan, conforming to the requirements of Specification E11.

5.2 *Extraction Apparatus*—The apparatus may be of the type shown in Fig. 1, or a Wiley-Richardson type, as shown in Fig. 2. The former type is more suitable for use with small electric hot plates, while the latter is more suitable for use with oil or water baths. In either case, it shall be possible to control the temperature so that the rate of extraction can be regulated accurately.

5.3 Drying Dishes—The drying dishes shall be lightweight dishes, approximately 63.5 mm ($2\frac{1}{2}$ in.) in diameter and 38.1 mm ($1\frac{1}{2}$ in.) in height.

6. Preparation of Sample

6.1 *Precautions*—It is of utmost importance that extreme care shall be taken during the preparation of the sample for

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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.



FIG. 2 Wiley-Richardson Type Extraction Apparatus

50 mm.

extraction. The sample shall be drillings if possible; however, if not possible, other suitable means of producing particles equivalent to drillings shall be used. Drillings taken from a large molded product shall be truly representative of all sections of the part in proper proportions. The drills for sampling shall be kept sharp and so operated that no undue heating of the material shall occur which will tend to precure the material.

Note 2—If it is impracticable to obtain samples by drilling, the parts may be broken up with a lathe, planer, milling machine, or a suitable grinder. A mortar and pestle or a pebble mill is considered suitable as a grinder, provided no perceptible heating occurs during the grinding procedure. A sharp file or rasp may be used for procuring the sample where the size or shape of the part is such that no other method is suitable.

6.2 In any case, the particles of the sample shall be of the smallest size practicable, so that they will pass through the No. 40 sieve with the minimum of reworking or grinding. It is important in preparing the sample that the smallest possible volume shall be obtained for a unit weight of the material.

6.3 The sample shall be sieved through a No. 40 sieve and that part which will not pass through shall be reground and blended with the original material passing the sieve. After assembling the Nos. 40 and 140 sieves and the receiving pan, the sample shall be placed in the top sieve, the cover placed on, and the entire sample shall be resieved either by a mechanical sieve shaker or hand sieving. If the hand-sieving method is used, the sieve shall be rotated with slight tapping, the period of rotation being 5 min.

6.4 After sieving, the sample (that portion which has passed through the No. 40 sieve and has been retained on the No. 140 sieve) shall be placed immediately in an airtight container to prevent absorption of moisture by the powder and the consequent error in results.

7. Conditioning

7.1 Condition the test specimens at $23 \pm 2^{\circ}$ C (73.4 \pm 3.6°F) and 50 \pm 10 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D618 for those tests where conditioning is required. In cases of disagreement, the tolerances shall be 1°C (1.8°F) and ± 5 % relative humidity.

7.2 Test Conditions—Conduct tests in the standard laboratory atmosphere of $23 \pm 2^{\circ}$ C (73.4 \pm 3.6°F) and 50 \pm 5% relative humidity, unless otherwise specified in the test methods or in this test method. In cases of disagreements, the tolerances shall be 1°C (1.8°F) and ± 2 % relative humidity.

8. Procedure

8.1 *Precautions*—Since the method is capable of exposing the user to potentially unhealthy fumes of volatile organic chemicals, notably acetone, such exposure shall be kept within strict conformity of applicable established OSHA Permissible Exposure Limits (PEL) and Time Weighted Average (TWA) rules and other available guidance protocols for any of the organic solvents used. Exposure guidelines should be available in the Material Safety Data Sheet, Section 8, Exposure Control / Personal Protection. Typical safety measures include the use of engineering controls, fully operating and properly adjusted