



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](#)

[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 \(apparent resin content of material in the un moulded state\)](#)

3. Terminology

3.1 All definitions are in accordance with Terminology [D883](#).

¹ This test method is under the jurisdiction of ASTM Committee [D20](#) on Plastics and is the direct responsibility of Subcommittee [D20.70](#) on Analytical Methods.

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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.

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](#).

5. Apparatus

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

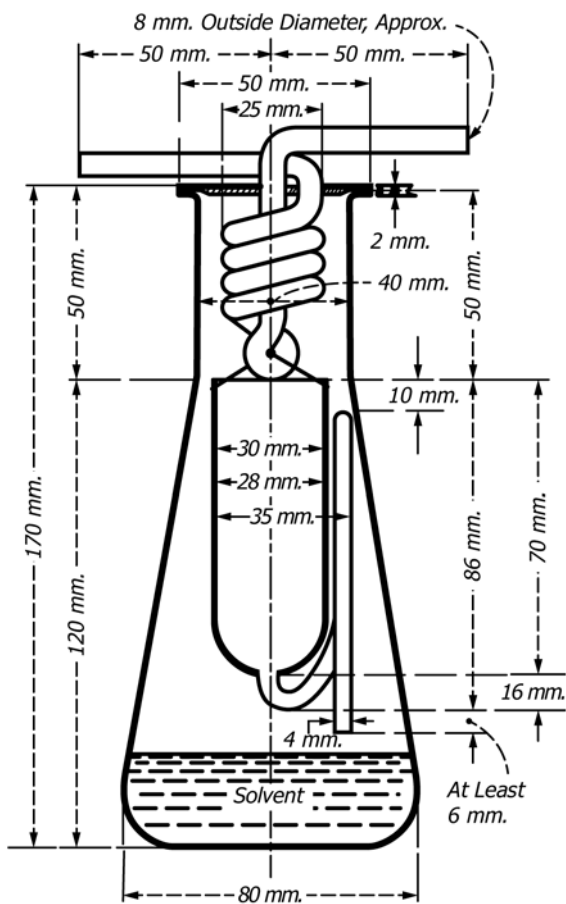
5.2 *Extraction Apparatus*—The apparatus may be of the type shown in [Fig. 1](#), or a Wiley-Richardson type, as shown in [Fig. 2](#). The former type is more suitable for use with small electric hot plates, while the latter is more suitable for use with oil or water baths. In either case, it shall be 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 (2½ in.) in diameter and 38.1 mm (1½ 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

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



(All dimensions in millimetres.)

FIG. 1 Extraction Apparatus

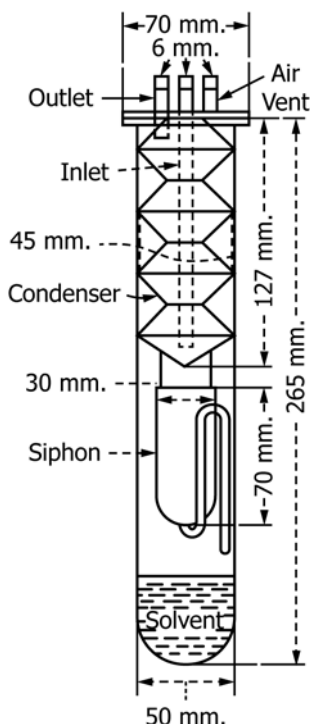


FIG. 2 Wiley-Richardson Type Extraction Apparatus

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 preclude 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 $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) 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 $\pm 5\%$ relative humidity.

7.2 Test Conditions—Conduct tests in the standard laboratory atmosphere of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) 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 $\pm 2\%$ 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

ventilation and personal protective equipment, such as hand, eye and breathing protection, along with reliable monitoring and confirmation of compliance with designated OSHA and other applicable exposure limits.

8.2 *Extraction*—The extraction procedure shall be carried out in triplicate. Accurately weigh a 3.000-g portion of the powdered sample into a tared, acid-hardened open-texture quantitative filter paper, 12.5 to 15 cm in diameter, or into a standard, single-thickness extraction thimble, 80 by 22 mm, trimmed if necessary. After folding over the thimble or filter paper containing the sample so that none of the powder can float out, place it in a desiccator until ready to insert in the siphon.

8.3 Press the filter paper or thimble containing the weighed sample into the siphon in such a way that the outlet of the bottom is not plugged. Place the condenser and the siphon in the extraction tube and add 50 mL of cp acetone. Start the water through the condenser and adjust the heat (**Note 3**) so that the siphon fills and empties between 15 and 20 times/h. This rate shall be carefully maintained, and the sample shall be extracted for 4 h. After the siphon empties, remove the flask and pour the contents into an individually weighed dish. Wash the flask three times with the smallest possible quantity of acetone, using a wash bottle, and add the washings to the extracted liquid in the dish.

NOTE 3—If an oil or water bath is used for heating, the height of the liquid in the bath should not come above 2.5 cm (1 in.) below the highest level of the acetone in the siphon before the siphon starts to discharge.

8.4 *Drying*—Place the dish in a well-ventilated drying chamber, maintained at $50 \pm 2^\circ\text{C}$ and dry the sample to constant weight. Between dryings, all dishes containing the residue shall be kept in a desiccator to prevent the absorption of moisture.

8.5 It is very important that the specified temperature shall be maintained, otherwise consistent results cannot be obtained between different laboratories.

NOTE 4—An electrically heated oven should not be used unless it is exceedingly well ventilated, as the acetone fumes are likely to come in contact with the heated coils and cause an explosion.

9. Calculation

9.1 Calculate the percentage of acetone-extractable matter in the specimen as follows:

$$\text{Acetone - extractable matter, \%} = [(W - D)/S] \times 100 \quad (1)$$

where:

W = weight of the dish and extract, g,

D = weight of the dish, g, and

S = weight of original sample, g.

10. Report

10.1 Report the following information:

10.1.1 Percentage of acetone-extractable matter for each sample, and

10.1.2 Average percentage of acetone-extractable matter for the three samples.

11. Precision and Bias³

11.1 **Table 1** is based on an interlaboratory study in 1989 in accordance with Practice **E691**, involving six materials tested by six laboratories.

11.2 Since no absolute method is available for comparison, no statement of bias can be presented for this test method.

12. Keywords

12.1 acetone extraction; cure; laminated plastic; molded plastic; phenolic resin; plasticizers

³ Supporting data are available from ASTM International Headquarters. Request RR:D20-1169.

TABLE 1 Precision Statistics

Values in the Units of Extracted %					
Material	Average	s_r^A	s_R^B	r^C	R^D
A	.3916	.08725	.2244	.24	.63
B	.5044	.0951	.2220	.27	.62
C	.5406	.1590	.2223	.45	.62
D	.5922	.2059	.2361	.58	.66
E	4.7628	.2142	1.2449	.60	3.49
F	4.6022	.2678	1.1051	.75	3.09

^A s_r is the within-laboratory standard deviation of the average.

^B s_R is the between-laboratories standard deviation of the average.

^C $r = 2.8 s_r$

^D $R = 2.8 s_R$

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D494 - 04(2010)^{e1}) that may impact the use of this standard. (September 1, 2011)

(1) Revised the relative humidity tolerance in 7.1 to be consistent with Practice D618.

Committee D20 has identified the location of selected changes to this standard since the last issue (D494 - 04) that may impact the use of this standard. (July 1, 2010)

(1) Added the ISO equivalency statement as Note 1.

(2) Removed non-mandatory language in sections 4.1, 5.2, 6.1, and 8.1.

(3) Changed Section 6.2 to Note 2.

(4) Renumbered Notes in text and renumbered text outline.

(5) Changed text immediately following section 8.5 to Note 4.

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