



Designation: **D4690 – 99 (Reapproved 2005) D4690 – 12**

Standard Specification for Urea-Formaldehyde Resin Adhesives¹

This standard is issued under the fixed designation D4690; 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 specification covers three types of urea-formaldehyde thermosetting resin adhesives suitable for use on wood, wood-based substrates, or plastic laminates.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information only.

1.3 The requirements for the adhesive are based on tests of physical properties including viscosity, density, nonvolatile content, pH, insoluble matter, amylaceous matter, mixing properties, working life, and on strength properties, including dry shear strength in lumber-type joints, dry and wet shear strength in plywood-type joints, and all strength tests at the end of the storage life certified by the manufacturer.

1.4 The following safety hazards caveat pertains only to the test method portion, Sections 88 through 113, of this specification: *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

[B417 Test Method for Apparent Density of Non-Free-Flowing Metal Powders Using the Carney Funnel](#)

[D905 Test Method for Strength Properties of Adhesive Bonds in Shear by Compression Loading](#)

[D906 Test Method for Strength Properties of Adhesives in Plywood Type Construction in Shear by Tension Loading](#)

[D907 Terminology of Adhesives](#)

[D1084 Test Methods for Viscosity of Adhesives](#) [ASTM D4690-12](#)

[D1489 Test Method for Nonvolatile Content of Aqueous Adhesives](#) [06-477a-9ef7-731bb82c9937/astm-d4690-12](#)

[D1490 Test Method for Nonvolatile Content of Urea-Formaldehyde Resin Solutions](#)

[D1583 Test Method for Hydrogen Ion Concentration of Dry Adhesive Films](#)

[D1875 Test Method for Density of Adhesives in Fluid Form](#)

[D2556 Test Method for Apparent Viscosity of Adhesives Having Shear-Rate-Dependent Flow Properties](#)

[D5266 Practice for Estimating the Percentage of Wood Failure in Adhesive Bonded Joints](#)

[E70 Test Method for pH of Aqueous Solutions With the Glass Electrode](#)

[E104 Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions](#)

2.2 Federal Specifications:³

[PPP-C-96 Cans, Metal, 28 Gage and Lighter](#)

[PPP-D-723 Drums, Fiber](#)

[PPP-D-729 Drums, Shipping and Storage, Steel, 55-gal
\(208-L\)](#)

¹ This specification is under the jurisdiction of ASTM Committee D14 on Adhesives and is the direct responsibility of Subcommittee D14.30 on Wood Adhesives. Current edition approved April 1, 2005/Oct. 1, 2012. Published April 2005/October 2012. Originally approved in 1987. Last previous edition approved in 1999/2005 as D4690 – 99/D4690 – 99 (2005). DOI: 10.1520/D4690-99R05-10.1520/D4690-12.

This specification is intended as a replacement for Federal Specification MMM-A-188c, Adhesive; Urea Resin Type (Liquid and Powder).

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.

³ Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, <http://dodssp.daps.dla.mil>.

2.3 *Federal Standard:*³

FED-STD-123 Marking for Shipment

2.4 *Military Standard:*³

MIL-STD-129 Marking for Shipment and Storage

3. Terminology

3.1 *Definitions*—Many terms in this specification are defined in Terminology **D907**.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *adhesive designation*—~~an adhesive that is manufactured by a unique combination of raw materials and manufacturing process that conforms to a given set of physical and performance properties and is identified by specific name, number, or alphanumeric designation.~~

3.2.1 *lot*—adhesive manufactured at one place from the same batch or blends of raw materials subjected to the same operations and conditions.

NOTE 1—In this specification, the maple block lamination in Test Method **D905** and the birch plywood construction in Test Method **D906** are described as assemblies.

4. Significance and Use

4.1 This specification provides testing procedures and specifies requirements to differentiate between the physical and adhesive bonding properties of the many commercial urea-formaldehyde thermosetting resin and catalyst systems.

4.2 Because of the diverse nature of products bonded with these adhesives, testing by this specification will only evaluate the adhesive under a given set of conditions. The physical properties of the adhesive are measured to provide information on certain handling and working properties. The adhesive-bonding properties are measured on maple block and birch plywood specimens, prepared and tested in accordance with Test Method **D905** and Test Method **D906**, respectively. The certification requirements have been based on knowledge within the industry of values that may be expected. Test Method **D905** and Test Method **D906** have a long history of use in many specifications.

4.3 The stress calculated by using this specification should not be used to predict failure nor should it be used for design stress in joints with different geometry or loading direction different from the test geometry.

5. Classification

5.1 Urea resin adhesive shall be furnished as one of the following types:

5.1.1 *Type A*—Powder with separate curing agent.

5.1.2 *Type B*—Powder with incorporated curing agent.

5.1.3 *Type C*—Liquid with separate curing agent.

NOTE 2—Type A, Type B, and Type C replace the Type I, Type II, and Type III classifications of Federal Specification MMM-A-188C, respectively, to prevent confusion with Type I (boil test) and Type II (cold-water soak) classifications used in existing ASTM standards for adhesives.

6. Test Requirements

6.1 To meet the requirements of this specification, the urea resin and adhesive mix shall be subjected to the tests listed in Section **10**. In addition, the adhesive shall meet the requirements of the tests described in Section **11** and as listed in **Table 1**.

6.2 To meet the storage-life requirements, an initial lot of the adhesive shall be tested in accordance with Sections **10** and **11**. It shall pass all test requirements after being stored under conditions and time specified by the manufacturer. Following this initial test, certification shall be based on submission from the manufacturer.

TABLE 1 Test Requirements

Test	Section Number	Test Requirement (Minimum)
Block shear (compression)		
—Dry at 24°C (75°F)	11.1	19 306 kPa (2800 psi)
—Dry at 24°C (75°F)	11.1	19 306 kPa (2800 psi)
Plywood shear (tension)		
—Dry at 24°C (75°F)	11.2.3.1	2 344 kPa (340 psi)
—Dry at 24°C (75°F)	11.2.3.1	2 344 kPa (340 psi)
—48-h soak	11.2.3.2	1 930 kPa (280 psi)
—48-h soak	11.2.3.2	1 930 kPa (280 psi)
Storage life	11.2.3.3	^A
Storage life	11.2.3.3	^A

^A A retained sample of the initial lot of the adhesive shall be tested and shall meet the requirements of all tests listed in **6.2** for the applicable type of adhesive after the storage life time certified by the manufacturer.

6.3 The curing agent for Types A and C shall readily disperse in the adhesive mixture when used in accordance with the manufacturer's recommendation.

6.4 The filler shall neither dissolve nor swell excessively in water, and shall not settle from the adhesive mixture during the working life.

7. Retest and Rejection

7.1 When a specimen fails at a load less than that specified, if the wood failure is 50 % or greater, that specimen shall be disregarded in computing the average. If more than one third of the test specimens for any one test condition are discarded for this reason, the test shall be repeated.

7.2 If the results of any initial test do not conform to the requirements prescribed in this specification, that test shall be repeated on an additional set of specimens made from the same lot of adhesive, each set of which shall conform to the requirements specified. If this set of specimens fails to meet the requirements, the lot shall be rejected.

TEST METHODS

8. Significance and Use

8.1 This specification provides testing procedures to differentiate between the physical and adhesive bonding properties of the many commercial urea-formaldehyde thermosetting resin and catalyst systems.

9. Sampling

9.1 Take a representative, 2-L (2-qt) sample of adhesive from the lot to be tested. Divide the sample into two equal portions and place each in a wide mouth, glass, 1-L (1-qt) container and seal tightly. Use one of the samples for tests of physical and adhesive bond properties (Sections 10 and 11), and use the other for testing storage life (11.2.3.3), and for any retesting that may be necessary under Section 7. Also, take sufficient quantity of curing agent to cure the adhesive samples and place in a separate, clean, dry container. Seal all sample containers with well-fitting, corrosion-resistant lids.

10. Physical Properties

10.1 Test the physical properties of adhesives by the following test methods:

10.1.1 *Viscosity:*

10.1.1.1 Measure the viscosity of the liquid urea resin or adhesive mixtures in accordance with Test Methods **D1084** or **D2556**.

10.1.1.2 *Temperature of Test*—Condition to, and maintain the liquid urea resin or adhesive mixture at $24 \pm 0.5^\circ\text{C}$ ($75 \pm 1^\circ\text{F}$) during the viscosity reading. Other temperatures may be used upon agreement between the contracting parties, but the $\pm 0.5^\circ\text{C}$ ($\pm 1^\circ\text{F}$) tolerance must be observed.

10.1.1.3 For Type C adhesives requiring the addition of a curing agent, make two viscosity measurements of the test adhesives (1) before the addition of curing agent, and (2) after the addition of the curing agent. The viscosity of urea resins following addition of curing agent to liquid resin is time-dependent. Make the measurement 10 min after mixing.

10.1.2 *Density:*

10.1.2.1 Measure the density of dry-powder adhesives or separate curing agents in accordance with Test Method **B417**, and report in grams per cubic centimetre (pounds per cubic foot). (See **Appendix X1**.)

10.1.2.2 Measure the density of Type C adhesives in accordance with Test Method **D1875** and report in grams per millilitre (pounds per gallon).

10.1.3 *Nonvolatiles:*

10.1.3.1 Determine the nonvolatile content of Type A and Type B from a 100-g mixture of adhesive and water (prepared by manufacturer's instructions) and test in accordance with Test Method **D1490**.

10.1.3.2 Determine the nonvolatile content of the uncatalyzed Type C liquid adhesive in accordance with Test Method **D1490**.

10.1.4 *pH of Cured Adhesive Film*—Determine the pH of the cured adhesive film in accordance with Test Method **D1583**.

10.1.5 *Insoluble Matter*—Determine the quantity of insoluble matter in Type B adhesives, including filler and any insoluble material, as a percentage of the nonvolatile constituents of the adhesive when determined as specified in 10.1.3.1. Weigh to the nearest milligram approximately 2 g of freshly mixed adhesive in a 100-mL beaker and add 50 mL of cold solution of 20 parts by volume of glacial acetic acid and 80 parts by volume of distilled water. Stir the mixture thoroughly for 1 min and then add 0.500 ± 0.001 g, acid-washed diatomaceous earth (see **Note 6**). Stir the mixture for 2 min more and then filter through a fritted-glass crucible of medium porosity with the aid of a vacuum. Transfer all residue to the filter with a 20 % acetic acid solution. Wash the residue on the filter with at least 50 mL of 20 % acetic acid and then with cold distilled water to remove the acid. Dry the crucible in an oven at $124 \pm 3^\circ\text{C}$ ($255 \pm 5^\circ\text{C}$) for 1 to 1½ h, cool in a desiccator, and weigh to the nearest milligram. The insoluble content, as a percentage of the nonvolatile content, is determined as follows:

$$\text{Percent insoluble} = (\text{Weight of residue} - 0.500) \times 100 / (\text{Weight of adhesive} \times \text{percent nonvolatile}) \quad (1)$$