Designation: D4690 – 12

Standard Specification for Urea-Formaldehyde Resin Adhesives[1]

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 8 through 11, 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

D1489 Test Method for Nonvolatile Content of Aqueous Adhesives

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)

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 *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.

[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 Oct. 1, 2012. Published October 2012. Originally approved in 1987. Last previous edition approved in 2005 as D4690 – 99 (2005). DOI: 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.

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.

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.

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)
Plywood shear (tension)		
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)
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.

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 ± 0.5°C (75 ± 1°F) during the viscosity reading. Other temperatures may be used upon agreement between the contracting parties, but the ±0.5°C (±1°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 ± 3°C (255 ± 5°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:

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

NOTE 3—"Filter-61" and "Celite Analytical Filter-Aid" have been found satisfactory. Diatomaceous earth is prepared by acid-wash, oven dried at 124 ± 3°F (155 ± 5°C) and then cooled in a desiccator.

10.1.6 *Amylaceous Matter in Type B Adhesive*—Add a drop of solution made from 5 g of iodine, 5 g of potassium iodide, and 100 mL of distilled water, to approximately 0.5 g of adhesive mix on a porcelain spot plate or watch glass. A darkening of the mixture denotes the presence of amylaceous matter.

10.1.7 *Mixing Properties and Working Life,* Mix approximately 200 g of adhesive (in accordance with the manufacturer's instruction) in a 400-mL beaker (Note 6) with a stirring rod or blade. Allow the beaker to stand without a cover in air at 24 ± 2.5°C (75 ± 5°F). If necessary, place in a water bath to obtain the proper temperature. Stir the adhesive frequently and note the time at which the adhesive is considered no longer spreadable. At the end of the working life, note whether the adhesive is in a homogeneous condition. In case of doubt as to whether the adhesive is spreadable, test the adhesive at 24 ± 2.5°C (75 ± 5°F) using a Brookfield Viscometer. Consider the adhesive to be at the end of its working life when it reaches a viscosity of 600 P (0.6 Pa·s). If the temperature of the bonding operation is 32.2°C (90°F) or higher, the above test may be conducted at 32.2°C (90°F).

NOTE 4—A disposable beaker or a 473-mL (16-oz) plastic cup may be used.

11. Adhesive-Bonding Properties

11.1 *Block-Shear Strength (Compression) Tests:*

11.1.1 Prepare and test specimens in accordance with Test Method D905. Use the adhesive manufacturer's instructions for conditions and procedures for preparing and spreading the adhesive, and assembling, pressing, and curing of the assembly. Use a two-week curing period unless otherwise instructed by the adhesive manufacturer.

11.1.2 *Number of Specimens*—Test 20 specimens representing at least four assemblies.

11.1.3 *Dry Shear Test*—Condition one test group of 20 specimens to 10 to 12 % moisture content at 24 ± 1°C (75 ± 2°F) and test in accordance with Test Method D905. Apply the load with a continuous motion of the movable loading head at a rate of 12.7 mm (0.5 in.)/min (±10 %).

NOTE 5—To obtain the desired moisture content refer to Table 3-4 on page 3-8 of the Wood Handbook.[4] Additional information is available in Practice E104. Constant temperature and relative humidity cabinets are available from laboratory supply companies.

11.1.4 *Calculation*—Calculate the shear stress at failure in kilopascals per square inch by dividing the specimen's breaking load by bond line area measured to the nearest 6.5 mm^2 (0.01 $in.^2$).

11.2 *Plywood Shear Tests (Tension):*

11.2.1 Prepare and test specimens in accordance with Test Method D906. Use the adhesive manufacturer's instructions for conditions and procedures for preparing and spreading the adhesive, and for assembling, pressing, and curing of the assembly. Use a two-week conditioning period unless otherwise instructed by the adhesive manufacturer.

11.2.2 *Number of Specimens:*

11.2.2.1 *Dry Tests*—Prepare 20 specimens from two assemblies. Test in accordance with 11.2.3.1.

11.2.2.2 *Wet Tests*—Prepare 20 specimens from two panels. Test in accordance with 11.2.3.2.

11.2.3 *Exposure Conditions and Treatments:*

11.2.3.1 *Dry Shear at* 24°C (75°F)—Condition the specimens to 10 to 12 % moisture content at 24 ± 1°C (75 ± 2°F) and test in accordance with Test Method D906.

11.2.3.2 *Forty-Eight-Hour Soak*—Immerse the test specimens in water 24 ± 1°C (75 ± 2°F) and soak them for 48 h ±

[4] "Moisture Content of Wood in Equilibrium with Dry-Bulb Temperature and Relative Humidity," *Wood Handbook*, No. 72, Forest Products Laboratory, United States Department of Agriculture, Revised August 1974, Table 3–4, pp. 3–8.

15 min. Remove the specimens from the water and test them immediately as specified in Test Method D906. Report individual and average test values.

11.2.3.3 *Storage Life,* Store the 1-L sample of adhesive collected in Section 9 at 24 ± 1°C (75± 2°F) for the period of time indicated by the manufacturer as the storage life of the adhesive. After storage, test for the requirements listed in Table 1. Report the length of the storage period.

NOTE 6—The storage life of adhesives will be reduced at an unacceptable rate at temperatures above 27°C (80°F).

12. Report

12.1 Report values for the physical properties tested in Section 10.

12.1.1 Report the temperature at which working life and viscosity were measured, that is either 23°C (73°F) or 32°C (90°F).

12.2 For the adhesive-bond test, Section 11.1, report individual and average test values, and the estimated percentages of wood failure for all specimens.

12.3 As a part of the report, include:

12.3.1 The manufacturer's recommended allowable storage period.

12.3.2 Mix-ratio used if the adhesive is a two-or-more-component system.

12.3.3 Certification by the manufacturer that the adhesive system meets the requirements of 6.3 and 6.4 as applicable.

12.3.4 Certification by the manufacturer that the adhesive passes all tests at the end of the recommended allowable storage period.

13. Precision and Bias

13.1 The precision and bias of the test methods measuring block shear strength (compression) and plywood shear strength are essentially as specified in Test Methods D905 and D906.

13.2 The precision and bias of Test Methods D1084, D1490, D1583, D1875 and D2556 will be determined as resources become available. The importance of Precision and Bias is recognized and that information will be gathered as time and personnel allow for it.

OTHER REQUIREMENTS

NOTE 7—The requirements mandated by the Department of Defense (DOD) have not been revised since publication of this specification. It is recommended that any manufacturer certifying to this specification consult with DOD to determine if any regulations have been added, revised, or deleted since the original publishing date.

14. Labeling

14.1 Include as part of the label, or advise consultation of, data sheet for the following:

14.1.1 An indication as to whether the adhesive is ready to use or if a curing agent must be added, along with any special instructions needed.

14.1.2 An expiration date for storage life.

15. Supplemental Government Requirements

15.1 *Packaging and Packing:*

15.1.1 *Preparation for Delivery*—Packaging and packing is Level A, B, or commercial as specified.

15.1.2 Unless otherwise specified, when Level A or B is specified, packaging and packing should be in accordance with the following criteria:

15.1.2.1 Metal containers conforming to PPP-C-96, Type V, class 2 for quantities of 50 lb (22.7 kg), maximum.

15.1.2.2 Fiber drums conforming to PPP-D-723, Type II, grade A or Type III, grade A for Level A, or fiber drums conforming to PPP-D-723, Type I, grade A for Level B, for quantities of 90.9 kg (200 lb), maximum.

15.1.2.3 Metal drums conforming to PPP-C-729, Type II or Type IV for quantities of 181.4 kg (400 lb), maximum.

15.2 *Containers:*

15.2.1 Containers should be uniform in shape and size, with the necessary protection required. Containers should contain quantities in accordance with the applicable container specification. All fiber drums shall be furnished with a 0.1-mm (0.004-in.) thick polyethylene liner, properly heat sealed.

15.2.2 Drums, as specified in PPP-D-723 or PPP-D-729, will require no overpacking.

15.3 *Shipment and Delivery*—Unless otherwise specified, when Level C is specified, the adhesive, 113 g to 136 kg (4 oz to 300 lb), should be packaged in containers to offer adequate protection against corrosion, deterioration and damage during shipment, handling and storage and should be packed to assure safe delivery to its destination when properly transported by any common carrier in conformance to requirements by Uniform Freight Classification or National Motor Freight Classification.

15.4 *Marking:*

15.4.1 Marking should be as specified in the contract or purchase order. Interior packages and shipping containers should be marked in accordance with Fed-Std-123 for civilian agencies or in accordance with MIL-STD-129 for military agencies.

15.4.2 Special markings when specified, in contracts, purchase orders, or by the contracting officer shall include:

15.4.2.1 Manufacturer's name, product code designation, and batch or lot number, and the national stock number.

15.4.2.2 Date of manufacture of product and expiration date.

15.4.2.3 Special-handling instructions during product transfer.

15.4.2.4 Special precautions related to toxicity, flammability or to any information pertinent to the proper handling and storage of the product, for example, manufacturer's storage temperature range.

16. Packing and Marking Requirements for Nongovernmental Users

16.1 The following sections shall apply to nongovernmental users of this specification, subject to the given exceptions: 15.3 (except for reference to Level C), 15.4.2 (whether or not specified), 15.4.2.1 (except for the national stock number), 15.4.2.2, 15.4.2.3 and 15.4.2.4.

17. Keywords

17.1 adhesives; density; insoluble matter; nonvolatile content; urea formaldehyde; viscosity

APPENDIX

(Nonmandatory Information)

X1. ADDITIONAL INFORMATION

X1.1 Test Method B417 covers the use of a standard funnel that feeds powder into a density cup. The bottom of the funnel and the top of the cup are 1.0 in. apart. A standard wire can be used to assist feeding of the powder through the funnel orifice until the cup overflows. After leveling the powder in the cup, the density is determined. Care should be taken not to jar the apparatus and effect compaction by vibration.