Designation: D 4689 – 99

Standard Specification for Adhesive, Casein-Type[1]

This standard is issued under the fixed designation D 4689; 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 casein and other protein-blend adhesives for use adhering wood to wood. The adhesive type covered by this specification is a dry powder or granular product comprising a mixture of casein, or casein and other protein source, with the necessary dry chemicals to effect solution when the mixture is added to water.

NOTE 1—Although the term *casein adhesive* is used throughout this specification, historically, the adhesive subclass described in 1.1 has been known as casein glue. See Terminology D 907, *adhesive types.*

1.2 *Limitations*—Cooked casein adhesives are not covered by this specification.

1.3 The values stated in inch-pound units are to be regarded as the standard.

1.4 The following safety hazards caveat pertains only to the test method portion, Sections 8 through 16, 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:*

D 899 Test Method for Applied Weight per Unit Area of Liquid Adhesive[2]

D 905 Test Method for Strength Properties of Adhesive Bonds in Shear by Compression Loading[2]

D 906 Test Method for Strength Properties of Adhesives in Plywood Type Construction in Shear by Tension Loading[2]

D 907 Terminology of Adhesives[2]

D 1084 Test Methods for Viscosity of Adhesives[2]

D 2556 Test Method for Apparent Viscosity of Adhesives Having Shear-Rate-Dependent Flow Properties[2]

D 5266 Practice for Estimating the Percentage of Wood Failure in Adhesive Bonded Joints[2]

E 104 Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions[3]

2.2 *Other Standards:*[4]

MIL-STD-129—Marking for Shipment and Storage

FED-STD-123—Marking for Shipment

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)

3. Terminology

3.1 *Definitions*—Many terms in this specification are defined in Terminology D 907.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *adhesive designation, n*—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 a specific name, number, or alphanumeric designation.

3.2.2 *lot, n*—adhesive manufactured at one place from the same batch or blends of raw materials subjected to the same operation and conditions.

NOTE 2—In this specification, the maple block lamination in Test Method D 905 and the birch plywood construction in Test Method D 906 are described as *assemblies.*

4. Significance and Use

4.1 This specification provides testing procedures and specifies requirements to differentiate the physical, adhesive, and durability properties of commercially available casein and casein-protein adhesives. The two classes are defined by water resistance. Selection of class is left to the choice of the product manufacturer and the consumer, based on measures used by each to protect the bond line from moisture degradation.

NOTE 3—Because of strict EPA requirements as to disposal of waste

[1] This specification is under the jurisdiction of ASTM Committee D-14 on Adhesives and is the direct responsibility of Subcommittee D 14.30 on Wood Adhesives.

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[2] *Annual Book of ASTM Standards,* Vol 15.06.

[3] *Annual Book of ASTM Standards,* Vol 11.03.

[4] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

TABLE 1 Test Requirements

Test	Section Number	Minimum Test Requirement	Test Required	
			Class B	Class C
Working life	6.1	5 h	yes	yes
Viscosity at 77 ± 0.5°F (25± 0.25°C)	6.1	3000 to 15 000 cp	yes	yes
Block shear (compression)				
Dry at 1 and 4 h	12.1	2800 psi (19 306 kPa)	yes	yes
Plywood (tension)	12.2	340 psi (2344 kPa)	yes	yes
Dry at 1 and 4 h[A]	12.2.3.1			
Soak, 48-h at 1 and 4 h[A]	12.2.3.2	140 psi (965 kPa)	yes	no
Storage life	14.	[B]	yes	yes

[A] One and four hours refers to age of adhesive after mix. See 6.2.1.
[B] A lot of the designated adhesive shall be tested for all the required tests after aging for the desired storage life time. The longest shelf life tested which passes all the minimum requirements shall be the storage life certified by the manufacturer.

from manufacturing and production facilities, it has become necessary for more than a decade to remove biocides from casein adhesives. Paint, varnish, and restrictive exposure conditions are protective measures commonly employed to protect the bond line against degradation due to mold and moisture.

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

5. Classification

5.1 For purposes of this specification, adhesives are classified on the basis of water resistance at two performance levels:

5.1.1 *Class B, Water Resistant*—An adhesive passing Class B test requirements, as listed in Table 1, is capable of producing sufficient adhesive-joint strength and durability and has sufficient water resistance to make the bonded products serviceable under conditions in which there will be occasional intermittent exposure to wet conditions or high humidity.

5.1.2 *Class C, Dry Use*—An adhesive passing Class C test requirements, as listed in Table 1, has sufficient adhesive-joint strength and durability under normal interior service conditions where the relative humidity is not high and does not fluctuate between wide limits.

6. Test Requirements

6.1 The test adhesives, when tested in accordance with Section 11, shall remain in the viscosity range of 3000 to 15 000 cP for a working life of at least 5 h, minimum.

NOTE 4—The viscosity requirement covers a 5-h span, although the "end-of-working-life" adhesive bond strength test is run at 4 h. This apparent conflict is necessary in order to assure that the mixed casein adhesive is not in a rapidly rising viscosity pattern at the end of the working life.

6.1.1 The working life shall be timed from the addition of the adhesive powder to the water.

6.1.2 The viscosity requirements shall not apply until the casein has dissolved and the adhesive is ready for use.

NOTE 5—Unless the manufacturer instructs otherwise, 20 min is the typical time period required, after the adhesive powder is added to the water, before the adhesive is ready for use.

6.2 To meet the adhesive bond requirements of the specification, the test adhesive shall be subjected to the tests described in Section 12 and shall meet the requirements listed on Table 1.

6.2.1 Two sets of adhesive bond tests are required, one to be initiated 1 h after mixing the adhesive with water, and one 4 h after mixing.

6.3 To meet the storage-life requirements, an initial lot of the adhesive shall be tested following the manufacturer's certified storage life, and shall pass all test requirements for the applicable adhesive classification. Following this initial test, certification for this requirement shall be based on submission from the manufacturer.

7. Retest and Rejection

7.1 When a specimen fails at a load less than that specified and the wood failure is at least 50 %, 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 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 the physical, adhesive, and durability properties of commercially available casein and casein-protein adhesives.

9. Apparatus

9.1 *Mechanical Stirrer or Mixer*, capable of speed in air of 5000 r/min for the preparation of the adhesive mix.[5]

9.2 Other apparatus required to conduct tests are covered in Test Methods D 899, D 905, D 906, D 1084 and D 2556.

10. Sampling

10.1 Take a 5-lb (2.3-kg) representative sample of the adhesive from the lot to be tested. Store samples in airtight containers and seal. For the initial testing of storage life only

[5] Satisfactory mixing may be obtained using the intermediate speed of the Thomas Three-Speed Stirrer No. 8585-M10 used with the stirring shaft, AHT No. 8633-T10, 9 in. (23 cm) long, and 1.5-in. (38-mm) diameter, four-blade propeller. Available from Thomas Scientific, Swedesboro, NJ 08085-0099, and other locations. Many other laboratory supply houses can provide this same mixer or its equivalent.

(6.4), divide the sample into two equal portions, packed as directed above. Use one portion for immediate testing and reserve the other for testing at the end of storage life.

11. Working Life

11.1 Prepare the adhesive for use in accordance with the manufacturer's instructions. Measure the time from the introduction of the powder into the water, and determine viscosity in a 400-mL beaker at 20 min and at 1, 2, 3, 4, and 5 h, in accordance with Test Method D 1084, Method B, with the following modifications:

11.1.1 Keep the adhesive at 77 ± 1°F (25 ± 0.5°C) for the duration of the test for working life.

11.1.2 Stir the adhesive with a mechanical stirrer for 1 min immediately before measuring the viscosity.

11.1.3 Allow the viscometer spindle to rotate for 30 s before recording the dial reading.

11.1.4 Using standard procedure for mixing a casein adhesive for testing, introduce the powder into the water with mechanical agitation, and continue the agitation for 1 min after the powder is dispersed. Turn off the mixer for approximately 15 to 20 min, depending on the manufacturer's instructions. Before the viscosity reading is taken, turn the mixer on again for 1 min. Then take the reading promptly.

11.1.5 Other temperatures may be used upon agreement between the contracting parties, but the ±1°F (±0.5°C) tolerance must be observed.

NOTE 6—Casein adhesives develop an exothermic reaction when added to water, so the water temperature must be 5 to 10°F (2.8 to 5.5°C) lower than the desired temperature of the liquid adhesive.

NOTE 7—Although casein adhesives and especially casein-soy blend adhesives have somewhat shear-rate-dependent flow properties, Test Method D 1084, Method B, is referenced for viscosity determinations rather than Test Method D 2556. The 1-min mixing time prior to reading a viscosity serves to break down the apparent viscosity build-up as the glue mix rests between readings. When more information is needed on the shear-rate-dependent flow properties of the adhesive, test the mix by Test Method D 2556.

12. Tests for Adhesive Bond

12.1 *Block Shear Strength (Compression)*:

12.1.1 Prepare the specimens in accordance with Test Method D 905, using the adhesive manufacturer's instructions.

12.1.1.1 In the absence of specific instructions, use a spread rate of 60 lb/1000 ft^2 (0.19 g/$in.^2$) of bond line (see Test Method D 899). The mixed metric/English system has been deliberately used, since it is a standard in the industry for spread rate. Use 1 to 2-min open assembly (time required to apply adhesive), 15-min closed assembly, 200 psi (1379 kPa) pressure, 2-h press time, and a 1-week conditioning period.

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

12.1.3 *Dry Test*—Following the prescribed conditioning period for the adhesive being tested, bring one test group of 20 specimens to 10 to 12 % moisture content and test in accordance with the instructions in Test Method D 905. Apply the load through a self-aligning seal to ensure uniform lateral distribution of the load. Apply the load with a continuous motion of the movable loading head at a rate of 0.5 in. (12.7 mm)/min (±10 %).

NOTE 8—To obtain the desired moisture content refer to Table 3–4 on page 3–7 of the Wood Handbook.[6] Additional information is available in Practice E 104. Constant temperature and relative humidity cabinets are available from laboratory supply companies.

12.1.4 *Calculation*—Calculate the shear stress at failure in pounds per square inch or kilopascals, based on the specimen's breaking load and tested bond-line area, measured to the nearest 0.10 $in.^2$ (6.5 mm^2).

12.2 *Plywood Shear Tests*:

12.2.1 Prepare the specimens in accordance with Test Method D 906, using the adhesive manufacturer's instructions.

12.2.1.1 In the absence of specific instructions, use a spread rate of 60 lb/1000 ft^2 (0.19 g/$in.^2$) of bond line. The mixed metric-English system has been deliberately used since it is a standard in the industry for spread rate (see Test Method D 899). Use 4 to 5 min open assembly time, 10 to 15 min closed assembly time, 2 h press time at 200 psi (1379 kPa), and a 1-week conditioning period.

12.2.2 *Number of Specimens*:

12.2.2.1 *Class B Tests*—Prepare 30 specimens, 10 from each of three assemblies. Separate the specimens randomly into 2 groups of 15 specimens each. Use one group for the dry test (12.2.3.1) and one group for the soak test (12.2.3.2).

12.2.2.2 *Class C Tests*—Prepare 20 specimens, 10 from each of two assemblies.

12.2.3 *Exposure Conditions and Treatments*:

12.2.3.1 *Dry Test at* 75°F (24°C)—Following the prescribed aging period and cutting of the test specimens, bring specimens (15 specimens for Class B, and 20 specimens for Class C) to 8± 1 % moisture content and 75 ± 2°F (24 ± 1°C) and test in accordance with Test Method D 906.

12.2.3.2 *Soak, 48-h*—Following the prescribed conditioning period and cutting of the test specimens, immerse the specimens in water at 75 ± 2°F (24 ± 1°C) and soak for 48 h, ±15 min. Remove from water and test immediately as specified in Test Method D 906, conducting the test in the wet state. Report individual and average test values. Dry the broken specimens to less than 8 % moisture content and note percentage of wood failure using Practice D 5266.

13. Storage Life

13.1 Store the sample in a tightly sealed container, at 75 ± 2°F (24 ± 1°C). Following the period of time given by the manufacturer for the storage life of the test adhesive. Test for compliance with the requirements listed in Table 1 for the applicable adhesive class.

14. Report

14.1 For the adhesive bond tests, Section 12, report the individual and average test values and the individual and average percentages of wood failure for the specimens.

14.2 As part of the report include:

14.2.1 Certification that the adhesive meets all the requirements of the specification given in Table 1.

[6] *Wood Handbook, Wood as an Engineering Material, FPL-GTR-113,* Forest Products Laboratory, United States Dept. of Agriculture, "Moisture Content of Wood in Equilibrium with Stated Dry-Bulb Temperature and Relative Humidity," revised 1999, Table 3–4, pp. 3–7.

14.2.2 The manufacturer's recommended allowable storage period and storage life certification that adhesive complies with 6.4.

14.2.3 Dry powder-to-water ratio used for the test.

15. Precision and Bias

15.1 The precision and bias of the test methods included in this specification for measuring block shear strength (compression) and plywood shear strength (tension) are essentially as specified in Test Methods D 905 and D 906.

OTHER REQUIREMENTS

16. Instruction Sheet

16.1 A dated, coded, and titled instruction sheet that outlines instructions for use of the adhesive is to be supplied by the manufacturer.

17. Packing and Marking Requirements for Nongovernmental Users

17.1 The following sections shall apply to nongovernmental users of this specification, subject to the given exceptions and modifications: 18.3.1 (except for reference to Level C), 18.4.2 (whether or not specified), 18.4.2.1 (except for the national stock number), 18.4.2.2, 18.4.2.3, and 18.4.2.4.

18. Supplemental Government Requirements

NOTE 9—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 published date.

18.1 *Packaging and Packing*:

18.1.1 *Preparation for Delivery*—Packaging and packing shall be Level A, B, or commercial, as specified.

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

18.1.2.1 Metal containers shall conform to PPP-C-96, Type V, Class 2 for quantities of 50 lb (22.7 kg), maximum.

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

18.1.2.3 Metal drums shall conform to PPP-D-729, Type II or Type IV for quantities of 400 lb (81.8 kg), maximum.

18.2 *Containers*:

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

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

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

18.4 *Marking*:

18.4.1 Marking is as specified in the contract or purchase order. Interior packages and shipping containers shall be marked in accordance with Fed. Std. No. 123 for civil agencies or in accordance with MIL-STD-129 for military agencies.

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

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

18.4.2.2 Date of manufacture of product, and expiration date.

18.4.2.3 Special handling instructions, during product transfer.

18.4.2.4 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.