Designation: D4689 – 12

Standard Specification for Adhesive, Casein-Type[1]

This standard is issued under the fixed designation D4689; 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 D907, *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 standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.4 The following safety hazards caveat pertains only to the test method portion, Sections 8 through 13, 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]

D899 Practice for Applied Weight Per Unit Area of Liquid Adhesive

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

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

E104 Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions

2.2 *Other Standards:*[3]

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 D907.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *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 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 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.

[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.

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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.

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

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.

NOTE 3—Because of strict EPA requirements as to disposal of waste from manufacturing and production facilities, most if not all biocides were removed from casein adhesives. Paint, varnish, and restrictive exposure conditions are protective measures commonly employed now 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.[4]

9.2 Other apparatus required to conduct tests are covered in Practice D899, and Test Methods D905, D906, D1084 and D2556.

[4] 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.