

# ANIMAL GLUES: THEIR MANUFACTURE, TESTING, AND PREPARATION

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ANIMAL GLUES: THEIR MANUFACTURE,  
TESTING, AND PREPARATION

By

Forest Products Laboratory,<sup>1</sup> Forest Service  
U. S. Department of Agriculture

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Classes and Grades of Animal Glue

The term "animal glue" refers broadly to all gelatin-type glues derived from by-products of the meat-processing and tanning industries. The most common raw materials used are the hides, bones, and tendons of cattle. Other by-products of cattle and certain parts of other animals are also utilized.

Glue made from hides is generally of higher grade, according to accepted standards, than glue derived from bones and tendons. However, there is considerable variation in the grades of glue from hides as well as from the other sources. Glues for most uses, including woodworking, are commonly blends of two or more batches from the same stock or from different classes of stock. Source is important only insofar as it affects grade.

Each class of glue is sold in cake, flake, ground, pearl, shredded, and other forms, but the form of the glue is no reliable indication of quality. The chief difference between the various forms is in the quickness and convenience with which they can be put in solution. The finely divided forms absorb water more rapidly and can be dissolved more easily than the cake and flake forms.

The higher-grade glues, in flake form, are usually light in color and nearly transparent. Lower grade glues tend to be dark in color and opaque.

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<sup>1</sup>Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

Color and transparency, however, are not safe indications as to the quality, for low-grade glues are sometimes bleached. On the other hand, foreign substances, such as zinc white, chalk, etc., are frequently added to transparent glues to produce what are technically known as opaque glues. The added materials, while they apparently do no harm, do not increase the adhesive qualities. Aside from the fact that they give an inconspicuous glue line in a joint, the "opaque" or whitened glues have no apparent advantage over other glues of the same grade.

Glues are graded according to a variety of tests which will be described later. In the past no two manufacturers have tested their glues in exactly the same way, nor have they followed the same system of grades. The oldest known system of classification is that of Peter Cooper, a manufacturer whose product was early established in the American trade. His grades, arranged in order of value, are: A Extra, I Extra, 1, 1x moulding, 1x, 1-1/4, 1-3/8, 1-1/2, 1-5/8, 1-3/4, 1-7/8, and 2. Later the National Glue Manufacturers' Association (7)<sup>2</sup> adopted standard methods of testing and a uniform system of grades.

High-grade hide glues are used mostly in joints of thick stock; lower grade glues for veneer work.

#### Manufacture of Animal Glue

The process of making animal glue is, briefly, as follows (2): The stock, after certain preliminary treatments to remove foreign matter and to get it into suitable condition for rapid conversion, is heated in water. The collagen, a complex insoluble protein body present in the raw materials, is thus hydrolyzed into the soluble, jelly-forming substances constituting glue. The hydrolysis continues beyond the formation of these products and gives rise to compounds having little or no jelly forming power and therefore of slight value as adhesives. For this reason the liquor is drawn off after a certain period of heating and a fresh supply of water added for a second run. A number of successive "cooks" may be made, but the glue obtained from the first is generally of the highest grade. There are many and varied details in the steps above outlined, depending upon the kind of stock used and the plant in which the glue is made. Any detail of manufacture may be expected to have its effect upon the character of the resultant glue.

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<sup>2</sup>Figures in parentheses refer to references at the end of the discussion.

The solution of glue from the boiling kettles is next concentrated by boiling off the water in vacuum dryers until the percentage of glue in the solution is high enough to make a firm jelly on cooling. If too high a temperature is reached during the concentrating process, the quality of the glue may be lowered.

When sufficiently concentrated, the glue solution is cooled by refrigeration, either after being run into pans or as it is carried upon a traveling belt. As it cools it forms a jelly firm enough to handle. From the pans, the jelly is removed, sliced with wires or a knife, and placed upon screens to dry. If a belt is used, the jelly is formed in a continuous sheet; this is cut into sections and placed on screens as it travels along. The screens are then placed in a drying chamber and left until the glue is dry. The glue may easily be injured during the drying process if the temperature conditions are not properly controlled. The form of the glue when dry depends upon the shape in which it was placed upon the screens. If carefully sliced to the proper thickness, cakes of regular shape will be formed. Sheet glue from the belt breaks into thin, irregular pieces as it comes from the drying screens. It is then commonly run through a machine to break it into smaller pieces, in which shape it is shipped as flake glue. In this or any other form, the dried glue may subsequently be ground and sold as ground glue. By varying the method of handling the glue as it comes from the evaporators the pearl glues or the shredded glues are produced.

#### Testing Animal Glue

Marked improvements have been made over a period of years in the standardization of methods for grading animal glues for woodworking. The National Association of Glue Manufacturers has adopted a standard method of grading based upon tests of viscosity and jelly strength (7). In 1931 a federal specification was adopted for procurement of animal glues for woodworking. The grades set up in the federal specification are based on tests of viscosity and jelly strength (13). It includes also tests for moisture content, pH, foaming, odor, and keeping quality and specifies the minimum requirements, considered adaptable to glues for woodworking. The definitely established tests and specifications afford the user of animal glues a method of insuring uniformity in the glue and of securing a product suited to his operating needs.

The following methods for testing animal glue are taken from the federal specification (13).

### Viscosity

Solutions of the glue for viscosity and jelly strength determination shall be prepared as follows: Sufficient of the glue sample shall be taken (calculated from the apparent moisture content) to give 13.2 grams of dry glue and placed in a standard wide-mouthed bottle of 150 cc. capacity with an inside diameter of 59 mm., an outside diameter of 66 mm., and a height over all of 85 mm., and fitted with a No. 9 rubber stopper. To the glue shall be added enough distilled water to make a total of 106.8 grams of water, including that naturally contained in the glue. This will give an 11 percent solution based on approximately anhydrous glue. The glue and water shall be thoroughly mixed with a stirring rod and then allowed to stand at a temperature of 10° to 15° C. for at least 4 hours. The glue shall then be melted by bringing the samples to a temperature of 62° C. in a water bath in which the temperature of the water shall not exceed 70° C. The time required to bring the sample to 62° C. shall not exceed 15 minutes.

The viscosity determination shall be made in a standard jacketed pipette viscometer or other suitable viscometer capable of expressing viscosity in the absolute unit of "poise." The instrument must be equipped to hold the glue solution at approximately constant temperature. The temperature of the glue solution shall be 60° C. and the determination made as soon as the solution has uniformly reached the required temperature.

### Jelly Strength

The samples from the viscosity test shall be placed in the standard containers and used for jelly strength determinations. The glue solution should be free of air or foam. The samples shall be allowed to cool to approximately 45° C. and any water of condensation shall be mixed thoroughly with the solution. The samples shall then be placed in a chamber or bath at  $10 \pm 0.1^\circ$  C. for a period of 16 to 18 hours.

The determination of jelly strength shall be made on a Bloom gelometer adjusted to give 4 mm. depression and to deliver a load of 40 grams per second to the surface of the jelly. The test must be completed within 2 minutes from the removal of the sample from the cooling chamber.

### Apparent Moisture Content

The loss in weight of a 10-gram sample, previously reduced to a size that will pass a No. 4 mesh screen, placed in a well-ventilated oven and heated at  $105^\circ \pm 3^\circ$  C. for 16 hours, shall be not less than 9 nor more than 15 percent of the original weight of the glue.

### Neutrality

Portions of the solutions upon which the viscosity and jelly strength tests were made or other samples of equal concentration shall be used to determine the acidity or alkalinity of the glue. The acidity or alkalinity shall be determined in terms of pH and must be within the range of 5.5 to 7.5

### Foam

Portions of the samples used in the viscosity and jelly strength tests or other samples of equal concentration shall be used for a determination of the tendency to foam. Seventy-five cc. of the solution at 60° C. shall be placed in the standard container and agitated with a stirrer or propeller. The stirrer shall be placed in the lower one-half of the solution and the speed of the stirrer during test shall be between 13,000 and 15,000 r.p.m. The solution shall be agitated for 1 minute and then allowed to stand at room temperature for 10 minutes, at the end of which there shall be not more than 2.5 cm. foam.

### Odor and Keeping Qualities

A 50 cc. portion of the glue solution used in previous tests shall be placed in an uncovered container and held in an incubator at 37°-38° C. for 48 hours. At the end of this time it shall be free from any odor of decomposition.

### Grease, Ash, and Other Miscellaneous Materials

If for any reason it seems desirable, tests may also be made for ash and grease content and the presence of materials that give a fictitious value to a glue. The presence of more than 4 percent ash in clear glues and 10 percent in opaque (white) glues, or 3 percent of grease (determined by the Kissling or other satisfactory extraction method that shows total grease), or the presence of materials which increase the viscosity or jelly strength may be cause for rejection.

### Wood-Joint Tests

Wood-joint tests are not a part of the federal specification. They are not suitable for the grading of animal glues because more direct and accurate evidence of the physical properties of the glue is afforded by the

viscosity and jelly strength tests. Strong joints may be made with a number of grades of animal glue but different gluing conditions must be used according to the grade of the glue. If wood-joint tests are made with glues of different grades under a uniform set of gluing conditions, the grade of glue that gives the best results is the one best adapted to the particular gluing conditions used and the results are not an accurate measure of the inherent strengths of the grades tested (12).

Wood-joint tests are, however, of decided value in determining the combination of gluing conditions that should be used with any given grade of glue. The Forest Products Laboratory has adopted the following procedure for making the wood-joint test (11): Selected pieces of wood, usually sugar maple, are stored in an atmosphere at 30 percent relative humidity until they have reached an equilibrium moisture content with this condition. Matched pieces, approximately  $3/4$ " x 2.5" x 12", are carefully surfaced to insure smooth, plane, and parallel surfaces. The pieces are glued under controlled conditions of temperature, amount of glue spread, duration of assembly period, and amount of pressure applied. They are allowed to remain under pressure overnight and then returned to 30 percent relative humidity for seasoning. A seasoning period of at least a week is allowed between gluing and testing. The glued pieces are then cut into specimens and tested in shear. The unit pressure required to break the joint and the amount of wood fiber torn in breaking are measures of the quality of the joint. The effect of gluing conditions on joint strength is discussed in detail in U. S. Department of Agriculture Bulletin 1500 (11).

#### Durability of Animal Glued Joints

Animal glues are generally low in water resistance. Conventional shear-test specimens of yellow birch plywood (3-ply,  $3/16$ -inch plywood) glued with unmodified animal glue have been exposed under each of several repeating cycles. The cycles consisted of exposure for 14 days at a higher relative humidity or soaking in water for 2 days followed by 12 or 14 days at 30 percent relative humidity all at 80° F. Such cycles cause swelling and shrinking of the wood and subsequent mechanical stresses on the joints. At some of the higher relative humidity conditions softening and hydrolysis of the glue also occurs. The exposure cycles were as follows:

Test	Type of exposure cycle	First stage		Second stage	
		Time	Relative humidity	Time	Relative humidity
		Days	Percent	Days	Percent
1	Continuous (controls)	.....	30	.....	.....
2	Alternating	14	60	14	30
3	.....do.....	14	80	14	30
4	.....do.....	14	90	14	30
5	.....do.....	14	97	14	30
6	.....do.....	*2	*	12	30

\*Soaked in water at room temperature.

Specimens subjected to Test No. 6 separated completely either in the first cycle or in the drying that immediately followed. Specimens exposed to Test No. 5 showed at the end of the first complete cycle an average joint strength of less than 40 percent of the original. All specimens failed before the end of the fourth cycle or in somewhat less than 16 weeks. A relative humidity of 97 percent is sufficiently high to permit development of molds and to bring the moisture content of the wood to about 28 percent during the "wet half" of the cycle.

Specimens exposed to Test No. 4 failed much less rapidly than did those exposed to Test No. 5. Animal-glued joints retained 75 percent or more of the initial strength in the 90-30 percent relative humidity cycle over a 6-month period, and 1 animal glue retained 96 percent of its initial strength for 12 months in this exposure. After that period all such glues lost strength rapidly in this cyclic exposure, retaining little or no strength after 18 months. Moisture content of the wood during the "wet half" of this cycle probably reached about 20 percent.

Two sets of animal-glued specimens retained 75 percent or more of their initial strength over a 4-year period of exposure to the 80-30 percent relative humidity cycle (Test No. 3). In this exposure the wood approaches 17 percent moisture content during the high humidity phase of the cycle. Furniture and other products glued with animal glues often serve satisfactorily in spite of occasional exposure to relative humidities in excess of 80 percent but in those cases protection afforded by the finish may prevent the moisture content of the wood from reaching equilibrium values, particularly if the exposure to dampness is not prolonged.

In Tests Nos. 1 and 2, no evidence of significant loss in strength could be detected during the 160 weeks that the tests were in progress. In another similar group of tests, specimens were continuously exposed for 5 years at 80° F. and 65 percent relative humidity without showing any significant losses in strength. Test No. 2 approximates the changes in moisture content that can be expected in interior woodwork in normal use in heated buildings in the northern part of the United States (8). In this type of service, properly designed and well-made joints of animal glue should prove permanently durable, and experimental evidence, other than these tests, supports that belief (3).

#### Modifications of Animal Glue for Woodworking

A considerable amount of study over a period of years has been devoted to development of methods for improving the water and moisture resistance of animal glues and their resistance to attack by micro-organisms since lack of permanence under moist conditions is one of the important limitations on the wider use of animal glue. Methods have been of two types. In one type certain preservatives, such as chlorinated phenol derivatives and organic mercurial compounds, have been incorporated in the glue mixture in the pot or spreader. When present in sufficient quantities such preservatives might be expected to reduce decomposition of the glue in the factory due to attack by micro-organisms while in the pot or spreader and to reduce similar damage to the glue in the joint in service. However, such preservatives would not be expected to improve the resistance of the glue to chemical hydrolysis in service, a reaction that is equally as likely and serious as micro-organic attack.

Efforts to improve the resistance to moisture and hydrolysis have included the addition of certain other agents to the glue mixture or the separate addition to the opposite wood surface of an agent that will react with the glue when the joint is assembled. Examples of such agents are tanning materials that give some increased moisture resistance, when added directly to the glue solution, but result in inconveniently limiting the pot life. Formulations (4) have been developed, however, that provide a pot life of 5 or 6 hours. Formaldehyde or some of its derivatives are often used for separate applications. Limited tests have indicated that some of the separately applied agents produce limited improvements in moisture resistance of animal glue joints in birch plywood but that after prolonged exposures under high humidity conditions the joints decrease in strength to approximately the same level as the untreated animal glue joints.

## Selection and Preparation of Animal Glue for Use

Making strong joints depends primarily upon having the proper correlation of gluing pressure and glue consistency at the moment pressure is applied. With animal glue solutions, the consistency depends on the cooling and drying effects. For the first few minutes after an animal glue has been spread on the wood the cooling effect is much more important than the drying and this temperature-viscosity relationship varies with the grade and with the concentration of the glue solution. High-grade animal glues thicken to the proper pressing consistency quicker and at higher temperatures than do low-grade glues of equal concentration. Assuming glues of equal grade, one mixed with less water will thicken more rapidly than one mixed with a greater quantity of water.

Warm animal glues, as they normally exist in the spreader, are too thin for pressing and some thickening must occur before pressure is applied. The best consistency for pressing exists when the glue is thick enough to form short, thick strings, when touched with the finger but not thick enough to resist an imprint or a depression readily. The thickening time or assembly period is usually fixed by the operating conditions that dictate how much time shall elapse between spreading and pressing. The grade of glue and the proportion of water added in mixing become, therefore, the variables by which the manufacturer can fit the glue mixture to his operating conditions. When once established, the grade and proportion of water should be adhered to except as temperature changes in the gluing room or wood require a change in the mixture.

The following tabulation shows the grades, with the corresponding ratings for jelly strength and viscosity, as established by federal specifications for animal glue for woodworking (13):

<u>Grade</u>	<u>Jelly strength</u> (grams)	<u>Viscosity</u> minimum (millipoises)
V1	150-177	59
V2	207-236	72
J1	237-266	83
J2	299-330	100
S1	331-362	111
S2	362-394	125

As a general rule, the following division of grades is recommended for different uses and service conditions:

V1 and V2 for general use in veneering where assembly periods may be somewhat prolonged.

J1 and J2 for general use in joint work where assembly periods are short.

S1 and S2 for special use where a quick chilling glue may be required.

As a further guide in selecting the grade and concentration of glue for a particular job, the following schedule (11) will produce good joints when an animal glue equivalent to the J2 grade is mixed in the proportions of 1 - 2-1/4.

<u>Temperature of room and wood</u> (°F.)	<u>Assembly period</u> (Minutes)
70	1/2 to 1
80	3 to 5
90	10 to 18

When the assembly period is fixed by the operation and the temperature in the glue room rises, an adjustment must be made to accelerate the speed of thickening. This adjustment can usually be made most easily by mixing less water with the glue.

The above schedule of conditions can be met with a lower grade, as J1 for example, by mixing less water with the glue than recommended for the J2 grade. Likewise, a higher grade, as S1 or S2, may be made to conform by mixing more water with the glue. Within limits, therefore, a user may purchase a glue of higher rating and mix it with more water or one of lower rating and mix it with less water to fit a particular operation.

In preparing animal glue for use a number of precautions must be observed if satisfactory results are to be obtained. The proportion of glue and water should be varied to meet manufacturing conditions (11). When the right proportions have been worked out they should be adhered to consistently. The glue and the water should be weighed out whenever a batch is prepared. Clean, cold water should be used and the mixture thoroughly stirred at once to allow a uniform absorption of water by the dry glue and prevent the formation of lumps. The batch should then stand in a cool place until the glue is thoroughly water soaked and softened. The soaking may take only an hour or two, or it may take several hours, the

time depending upon the size of the particles. The glue should then be melted over a water bath at a temperature not higher than 150° F. High temperatures and long continued heating reduce the strength of glue solutions and are, therefore, to be avoided. The glue pot should be kept covered as much as possible in order to prevent the formation of a skin or scum over the glue surface.

Strict cleanliness of glue pots and apparatus and of the floors and tables of the glue room should be observed. Old glue soon becomes foul and affords a breeding place for the bacteria of decomposition, exposing the fresh batches to the constant danger of becoming contaminated. Glue pots should be washed every day and only enough glue for a day's run should be mixed at a time. If these simple, sanitary precautions are not observed, poor joints are more than likely to result.

In a brief review, it is not possible to cover thoroughly all the points touched upon, or even to mention many facts which the glue user should know. For more detailed treatment of the subject, with information on animal glues and other types as well, the reader is referred to the publications listed on page 11.

#### Liquid Glues

Most of the commercial liquid glues are made from fish stock -- heads, bones, skins, and swimming bladders -- although several brands of animal glue in liquid form are on the market. Liquid glue can be used cold and spread without any preparation whatsoever. Commercial liquid glues vary widely in strength; some are so weak as to be suitable only for use on paper or cardboard, while others are almost as strong as high-grade dry animal glue.

Liquid glues are used to a certain extent in joint work, but not of the highest class. They are used also for repair work and miscellaneous purposes. Liquid glues could be more generally recommended if the better grades could be readily and accurately distinguished by the user.

#### Testing Liquid Glues

Methods of testing liquid glues have not been standardized. Tests of joint strength on liquid glues have indicated that the very thin, watery liquid glues tend to produce weak joints, but no definitely conclusive

relation appears to exist between viscosity and joint strength. Tressler (10) has suggested the following tests for liquid glues from fish stock: Viscosity, ash content, drying properties, hygroscopicity, joint strength, color, permanence, and pH value. Jennings (6) holds that liquid fish glues should have the following properties: Be neutral to litmus, slightly acid to phenolphthalein, remain unaltered after one month's storage at 37°-38° C., set to a jelly between 7.5° and 10° C., be free from tackiness when exposed in film form at 25° C. to 80 percent relative humidity, and contain not more than 0.2 percent chlorides.

Since the importance or value of the various chemical and physical properties of liquid glues are not well established and since standardized test procedure for their determination is not available it is necessary to place more reliance on joint tests. The procedure described for making joint tests on animal glues is also applicable to liquid glues. Here again joint tests must be carefully made to be of value.

## References

- (1) ALEXANDER, J.  
1923. Glue and gelatin. Chem. Catalog Co., New York.
- (2) BOGUE, R. H.  
1922. The chemistry and technology of gelatin and glue. McGraw-Hill Book Co., New York.
- (3) BROUSE, DON  
1931. Age and strength of glue joints. Woodworking Industries, June, pp. 32, 33.
- (4) BROWNE, F. L.  
1933. A water-resistant animal glue. Forest Products Laboratory Report No. R40.
- (5) GILL, A. H.  
1925. A comparison of various tests upon glue. Indus. & Eng. Chem. 17, 297-8.
- (6) JENNINGS, D.  
1933. Quality of fish glues. Chem. Trade Jour. 92, 105-7.
- (7) NATIONAL ASSOCIATION OF GLUE MANUFACTURERS  
1930. Standard methods (revised) for determining viscosity and jelly strength of glue. Indus. & Eng. Chem., Anal. Ed. 2, 348-51.
- (8) PECK, E. C.  
1932. Moisture content of wood in dwellings. U. S. Dept. of Agr. Circ. 239.
- (9) RIDEAL, A.  
1926. Glue and glue testing. Scott, Greenwood & Son, London.
- (10) TRESSLER, D. K.  
1924. Examination of liquid glue. Indus. & Eng. Chem. 16, 943-5.
- (11) TRUAX, T. R.  
1929. The gluing of wood. U. S. Dept. of Agr. Bull. 1500.
- (12) \_\_\_\_\_, BROWNE, F. L., and BROUSE, DON  
1929. Significance of mechanical wood-joint tests for the selection of woodworking glues. Indus. & Eng. Chem. 21, 74-9.
- (13) UNITED STATES FEDERAL SPECIFICATIONS BOARD  
1945. Federal specification CG451, as amended, Feb. 17, 1945:  
"Glue, animal, woodworking."