



Methods for

Sampling and testing glues (bone, skin and fish glues)

Méthodes pour l'échantillonnage et l'essai des colles (colles à base d'os, de peau et de poisson)

Verfahren für Probenahme und Prüfen von Leimen (Knochen-, Fell- und Fischleimen)

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Foreword

This British Standard has been prepared under the direction of the Adhesives Standards Committee. It differs from the 1969 edition in that a second method for the determination of viscosity has been added using the American pipette; the method for the determination of melting point using indicator solution has been deleted; a method for determination of setting point and a second method for the determination of foam using a whisk have been added.

British Standard Methods for

Sampling and testing glues (bone, skin and fish glues)

0. Introduction

0.1 General. The physical and chemical properties of glue depend, first on the nature of the raw material, and secondly on the methods of processing. It is not possible to draw up any simple tests that will evaluate completely the quality of a glue, or its suitability for a particular use. If, however, the glue is made from a specific type of raw material by a usual method of manufacture, then the following tests provide indications of the behaviour of the glue in use, and may be taken as reliable criteria of quality.

The relative importance of the tests to be employed depends very largely on the purposes for which the glue is intended. For instance, the joinery trade is particularly interested in joint strength, the gummed paper and the carton manufacturer in viscosity and foam. It is not necessary to perform all the tests described on any one sample. In practice the user often knows from experience the most suitable type of glue for his purpose. The problem then reduces itself to checking those properties that are relevant.

Amongst these properties, the jelly strength or the viscosity or both are usually included as a general index of quality. The foam test is of interest when the glue is to be applied by special machines. The keeping quality test is intended simply as an index of the behaviour of the glue in practice, and is not to be taken as a substitute for a more complete bacteriological examination. The sulphur dioxide test is important when the glue is to be used for coloured paper or textiles. The chloride content is one index of the behaviour of glue under various atmospheric conditions. The joint strength in shear is an indication of whether the glue is sufficiently strong for joinery purposes.

For certain uses there are also specialized tests, which can be agreed between purchaser and vendor, that are not suitable for inclusion in the present standard.

Many of the physical tests (e.g. jelly strength, water absorption, foam) are admittedly of an empirical character, but if the methods are carefully followed consistent results are obtainable which will give useful information both to the manufacturer and to the user.

This British Standard describes methods for sampling and testing only and should not be used or quoted as a specification.

0.2 Importance of moisture content. The rapidity with which small samples of powdered glue change their moisture content, in response to changes in atmospheric humidity, emphasizes the need for care, after a sample has been taken, in order to keep the moisture content unchanged. The use of sealed waterproof containers is desirable.

If a consignment undergoes long delays in transit, the average moisture content may change between the times of sampling by producer and consumer. To avoid this causing unnecessary disputes, it is recommended that when jelly strengths or viscosities are reported, the results of moisture content tests should also be given, if an accurate comparison is required. Although the remaining tests listed in table 1 may also be slightly affected by changes in moisture content, the differences are not significant and it is not necessary to report moisture contents for them.

An approximate formula for the change in jelly strength (ΔB) for a change in moisture content (ΔM) is:

$$\Delta B = \frac{-2B_1 \Delta M}{100 - M_1}$$

where

B_1 is the jelly strength at a percentage moisture content M_1 .

This formula may be used to calculate the jelly strength of glue at an agreed moisture content, e.g. 15 %.

If M_1 is taken as 15 %, then $\Delta B = B/42.5$ for each 1 % change in moisture content, i.e. at 250 g Bloom, $\Delta B = 6$ g Bloom per 1 % change.

There is no satisfactory formula for the change of viscosity, $\Delta \eta$, with moisture content, ΔM , but a rough guide is given by $\Delta \eta = -2.6\eta_1 \frac{\Delta M}{100}$. This formula applies to 12.5 % solutions.

0.3 Sampling. The results of the analysis of glue carried out in accordance with this standard are limited in their practical use by the degree to which the 1 kg final laboratory sample represents the whole delivery. Guidance is therefore given in clause 4 on bulk sampling.

Warning note. The test methods in this British Standard do not necessarily detail all the precautions necessary to meet the requirements of the Health and Safety at Work etc. Act 1974. Attention should be paid to any appropriate safety precautions and the methods should be operated only by trained personnel.

1. Scope

This British Standard specifies the methods to be used for sampling and testing bone, skin and fish glues.

2. References

The titles of the standards publications referred to in this standard are listed on the inside back cover.

3. Definitions

For the purposes of this British Standard, the following definitions apply.

3.1 moisture content. The percentage loss in mass of the sample where a thin evenly distributed film of the glue is dried at 105 °C for 18 ± 1 h under standard conditions (see clause 6).

3.2 jelly strength. A measure in arbitrary units of the rigidity modulus of a gel prepared and matured under standard conditions (see clause 7).

4. Sampling

4.1 Initial selection of sample

4.1.1 Powdered, pearl, cube and granulated glue. The number of containers sampled should be fixed by agreement between purchaser and vendor. As a guide, it is suggested that the number sampled in each consignment should be that given in BS 5350 : Part E2. The containers should be selected from various parts of the consignment in such a manner that the widest representation is obtained (see BS 5350 : Part E2).

From containers of 50 kg or more of glue, take a sample weighing not less than 1 kg, when there is only one container, and an increment of not less than 500 g from each container when there is more than one; from containers of less than 50 kg of glue take a proportionate amount.

Take each increment by means of a sampling tube, scoop or similar tool to ensure that glue is taken from the top, middle and bottom of the container.

4.1.2 Slab and jelly glues in container. Follow the instructions in 4.1.1 concerning the number of containers to be sampled and take the same amount from each container; do not include any surface skin.

4.1.3 Liquid glue. Sample as for slab and jelly glue, mixing the contents of the container by stirring before sampling. For the accurate sampling of large vessels, suitable instruments are described in BS 5350 : Part E2.

4.2 Final selection of sample

4.2.1 General. Place the samples, taken as described in 4.1, in clean, dry air-tight non-absorbent containers. Leave them there until the reduced samples are taken.

Take one or more reduced samples, each weighing not less than 1 kg, as described in 4.2.2 to 4.2.4, and place them in clean, dry, air-tight, non-absorbent containers.

4.2.2 Powdered, pearl, cube and granulated glues. Mix the material thoroughly and take a reduced sample from the bulk.

4.2.3 Slab and jelly glue. Cut a representative piece from each slab or portion. Cut these pieces into smaller portions and reduce by mixing and quartering.

4.2.4 Liquid glue. Bulk the representative portions in a vessel, mix by stirring and take a reduced sample.

5. Preparation of laboratory sample

5.1 Reduction of solid (powdered, pearl, cube and granulated) glue sample. Grind the sample by hand in an iron mortar, or alternatively in a laboratory disintegrator,

of a type agreed between purchaser and vendor, down to a particle size of approximately 3 mm. If necessary, quarter this sample in the usual manner to bring the final mass to 500 g, taking care that a representative amount of all particle sizes is included in the quartering. Keep the powdered sample so obtained in an air-tight container, and use this sample for all the tests except the moisture content test (see clause 6).

NOTE 1. In view of the loss of moisture in grinding, it is necessary to take a separate sample for the moisture content test from the quantity obtained by the preliminary breaking up, before putting through the disintegrator. This small separate sample can then be powdered by hand in a pestle and mortar, or cut with scissors, and is suitable for the moisture content test. It is necessary to make adjustment for the moisture content of the mechanically ground sample by comparing with the hand ground sample, which should be taken as having the more reliable moisture content. By comparing the actual moisture content found on the hand ground sample with the moisture content of the mechanically ground sample, the necessary adjustment to be made to the mass of the glue to be used in the various tests can be found by calculation.

NOTE 2. It is important to put all samples immediately into an air-tight container because even a small change in the moisture content will affect the result of some of the tests; for example, an increase of 1 % moisture would result in a decrease of about 2.5 % in jelly strength when determined on the Bloom type gelometer.

5.2 Concentrations for solid glues. Table 1 summarizes the mass of glue and volume of water required for each of the tests described. It is preferable to weigh the specified quantity of the powdered sample for each test separately, rather than work from a large quantity of stock solution.

5.3 Method of dissolving solid glues. Weigh the powdered sample in a beaker and add the requisite amount of cold distilled water*, stirring with a thin metal or glass rod. Place a watch glass on the beaker and allow the sample to soak for 3 h at a temperature not exceeding 22 °C. Heat the beaker in a water bath adjusted to a maximum temperature of 70 °C for about 15 min, taking care that the final temperature of the solution when in the bath reaches, but does not exceed, 60 °C. During this heating period gently stir the solution with a rod. Take care that the glue is completely dissolved: this may be ascertained by lifting up the beaker and inspecting its contents through the bottom. When the sample has all dissolved, remove the beaker from the water bath.

Table 1. Glue concentrations

Clause	Test	Mass of glue	Volume of water
		g	ml
6	Moisture content	1	10
7	Jelly strength (see 7.5.2)	15 (7.5)	105 (105)
8	Comparison of jelly strength	5 to 10	50
9	Viscosity	15	105
10	Melting point	37.5	75
11	Setting point	37.5	75
12	Foam: method 1	5	50
	method 2	15	105
13	Keeping quality	5	20
14	pH	1	100
15	Grease	10	15
16	Joint strength in shear	10	15

*Water complying with the requirements of BS 3978 is suitable.

If the sample gives a jelly strength above 400 g Bloom, the jelly shall be prepared at a concentration of 6.66 %, and a note made of this in the report. The results on a 6.66 % solution are sometimes expressed as single Bloom and that on a 12.5 % solution as double Bloom.

5.4 Treatment of liquid and jelly glues. Determine the moisture content (see clause 6) and from this calculate the equivalent of cake glue, assuming that cake glue contains 15 % of water. In all the subsequent determinations weigh out this calculated equivalent in place of the quantity stated in table 1, and adjust the amount of water to be added correspondingly. Do not evaporate water from a liquid or jelly glue in order to bring it down to a stronger concentration for the test.

For the joint strength in shear test, however, it is not necessary to add water, unless specifically instructed to do so. Use the glue undiluted at an agreed temperature and make a note in the report.

6. Method for the determination of moisture content*

6.1 Principle. A weighed sample of the glue is maintained at 105 °C for 18 h and is then reweighed.

6.2 Apparatus. The following apparatus is required.

6.2.1 Stainless steel dish, flat bottomed, 70 mm in diameter and 15 mm high, weighing about 30 g, preferably fitted with an aluminium cover for use when cooling and weighing.

6.2.2 Oven, complying with the requirements of BS 2648, capable of being maintained at 105 ± 1 °C.

6.2.3 Waterbath.

6.3 Procedure. Weigh into the tared dish, to the nearest 0.01 g, about 1 g of the unground sample (see note 1 in 5.1).

NOTE 1. In preparing the sample for this test it is necessary to remember that glue, especially in the finely divided state, rapidly alters in moisture content according to the relative humidity of the air to which it is exposed. It is important, therefore, that the operations of crushing and sampling described in 5.1 should be carried out without delay and in surroundings which avoid excessive humidity or dryness. There should be no delay in transferring the sample to an air-tight container pending weighing out.

Add 10 ml of distilled water and allow the glue to soak. Place the dish on a warm water bath so that the glue is dissolved and a homogeneous solution obtained, and leave there until most of the water has evaporated, giving a uniformly thin film.

Transfer the dish to the oven and allow it to remain there for 18 ± 1 h, during which time the oven door shall not be opened. Remove the dish from the oven, allow it to cool in a desiccator and weigh it.

NOTE 2. It is essential that every precaution should be taken to prevent access of moisture to the dried film before and during weighing.

Carry out the determination in duplicate.

6.4 Expression of results. Calculate the moisture content as a percentage by mass using the equation.

$$\frac{M_0 - M_1}{M_0} \times 100$$

where

M_0 is the mass of original sample;

M_1 is the mass of the sample after drying.

Express the result as the mean of the two values obtained in the duplicate determination.

6.5 Test report. The report shall state the following:

- (a) a designation of the glue giving all information required for complete identification of the sample;
- (b) the result of the test including the individual values and any circumstances which may have affected the result;
- (c) the method used i.e. clause 6 of BS 647 : 1981.

7. Methods for the measurement of jelly strength

7.1 Principle. If there is no agreed sample for comparison, it is necessary to measure the jelly strength of the sample for test under arbitrary conditions. A suitable instrument measures the force necessary to give a 4 mm depression in a jelly of 12.5 % concentration by mass, matured for 16 h to 18 h at 10 °C, using a standard plunger (see 7.2.4). By convention, this force is expressed as a mass in grams.

7.2 Apparatus. The following apparatus is required.

7.2.1 Bottle, of internal diameter 59 ± 1 mm, approximately 85 mm high, having a capacity of approximately 155 ml†. A stopper of rubber or cork approximately 43 mm in diameter should fit snugly into the neck of the bottle and should be pierced centrally with an air vent about 2.5 mm in diameter.

7.2.2 Thermostatic bath, capable of being maintained at 65 ± 1 °C.

7.2.3 Thermostatic bath fitted with thermometer, totally enclosed, and capable of being maintained at 10 ± 0.1 °C. (Thermometer no. A20C/100 complying with the requirements of BS 593 : 1974 and calibrated after stabilization as described in appendix C of that standard is suitable.)

7.2.4 Plunger, made of suitable, stable polymer with diameter of 12.7 mm and edge radius 0.40 mm (figure 1).

7.2.5 Gel testing instrument, that enables the plunger to be brought just into contact with the surface of the gel and then measures the force required to depress it vertically to a depth of 4 ± 0.01 mm, either at a constant rate of loading not exceeding 40 g s⁻¹ or at a constant rate of penetration not exceeding 0.8 mm s⁻¹. (See appendix A for details of suitable instruments.)

7.3 Dissolving the sample. Dissolve 15 g of the sample in 105 ml of water in the wide neck bottle (7.2.1) in the manner specified in 5.3, closing the bottle with the stopper. To prevent caking, swirl the bottle vigorously to wet the glue completely. At this stage, take care not to build up more froth by excessive agitation than will collapse before the bottle is inserted in the bath (7.2.2) maintained at 65 ± 1 °C. Particular care is necessary to see that the glue is all dissolved and that the solution is homogeneous. Striations in the solution after swirling the bottle indicate the need for more prolonged agitation. The striations are best seen in a bright light. Place a finger over the perforation in the stopper and invert the bottle several

*By agreement between purchaser and vendor, a suitable method using infra-red heating may be used provided it can be shown that the results obtained are similar to those obtained by the method described in this clause.

†For information as to the availability, apply to the Central Enquiries Section, British Standards Institution, enclosing a stamped, addressed envelope for a reply.

times to mix in the water that has condensed on the walls of the bottle and the under-side of the stopper.

7.4 Chilling the solution. To prevent cracking, allow the bottle to cool for 15 min at room temperature, and then place in the thermostatic bath (7.2.3), maintained at $10.0 \pm 0.1^\circ\text{C}$, for not less than 16 h and not more than 18 h. Ensure that the platform of the thermostatic bath is horizontal and that the bottle stands evenly on it.

7.5 Procedure for determining jelly strength

NOTE. See appendix A for detailed instructions for particular instruments.

7.5.1 It is essential that the instrument stands perfectly level on a rigid support, that the plunger face is parallel to the gel surface, and that the direction of plunger travel is perpendicular to the gel surface.

7.5.2 Place the test bottle containing the gel on the platform of the instrument so that the centre of the gel is underneath the plunger. Proceed with the sequence of operations required for the particular type of instrument in use and record the force required to depress the plunger 4 mm into the gel, translated as the jelly strength of the sample in grams. If the jelly strength is above 400 g, repeat the test using a solution prepared by dissolving 7.5 g of the sample in 105 ml of water to give a solution concentration of 6.66 %.

7.6 Expression of results. Express the results to the nearest gram at the appropriate concentration.

7.7 Test report. The test report shall state the following:

- (a) a designation of the glue, giving all information required for complete identification of the sample;
- (b) the result of the test, the concentration of the solution and any circumstances which may have affected the result;

NOTE. The result of the test using a 6.66 % solution is sometimes termed 'single Bloom' and that using a 12.5 % solution 'double Bloom'.

- (c) the method and instrument used (e.g. clause 7 of BS 647 : 1981 — Bloom gelometer).

7.8 Checking the jelly strength determination.

The determination may be checked completely in three stages.

- (a) By measurement of the diameter of the gelometer plunger and the radius of curvature of its lower circular edge (see figure 1). A profilemeter is a satisfactory instrument for measuring the radius of curvature.

- (b) If the instrument used cannot be calibrated by direct loading of known masses, then it may be calibrated using a dummy Bloom device* (see figure 2).

- (c) By checking melting and chilling conditions on every occasion using samples standardized against reference samples. All standard samples require correction for moisture content variations if the measured moisture content differs by more than 0.4 % from the value at the time of standardization.

8. Method for the comparison of jelly strength

8.0 Introduction. If there is a sample agreed between purchaser and vendor, it is necessary to compare the jelly strength of the sample for test with that of the agreed sample under the same conditions of test.

Any suitable instrument† may be used for this purpose, provided that there is previous agreement on procedure, concentration and temperature. (See also the foreword.)

8.1 Principle. A solution of glue is prepared and allowed to gel for 16 h before comparison of jelly strength, in a test instrument, with the agreed sample.

8.2 Procedure. Weigh 5 g to 10 g, according to grade and use of the powdered sample (see clause 5) into a 150 ml beaker, and add 50 ml of cold distilled water‡ from a pipette. Dissolve the sample in the manner specified in 5.3.

Pour the glue solution immediately into a suitable cylindrical container and, after 2 min, put the lid on. Leave the container for 16 h in a bath of running cold water, or preferably in a thermostatic bath maintained at an agreed temperature, then remove the lid and compare the jelly with that of the agreed sample, treated in the same way.

NOTE. Provided that the procedure, concentration and temperature are agreed, an instrument can be used to give a numerical reading in cases where there is no reference sample.

8.3 Expression of results. Express the jelly strength as less than, equal to or greater than that of the agreed sample, as the case may be.

8.4 Test report. The test report shall state the following:

- (a) a designation of the glue, giving all information required for complete identification of the sample;
- (b) the result of the test, the instrument used and any circumstances which may have affected the result;
- (c) the method used i.e. clause 8 of BS 647 : 1981.

*Developed by the Gelatine and Glue Research Association (now the Gels and Protein Section of the British Food Manufacturing Research Association, Randalls Road, Leatherhead, Surrey). Appropriate references to the development and use of the dummy Bloom are:

Angus, J. and Culshaw, A. J. (1966) *Gelatine and Glues Research Association (GGRA) Bulletin* 17, No.2, 8-14.

Angus, J. Pratt, A. N. and Wainwright, F. W. (1961a) *GGRA Bulletin* 12, No. 3 13-15.

Angus, J. Wainwright, F. W. and Wootton, J. (1961b) *GGRA Bulletin* 12, No. 2, 15-17.

Fysh, D. and Ward, A. G. (1953). *GGRA Research Report* B7.

Wainwright, F. W. (1961). *GGRA Bulletin* 12, No. 3, 16-18.

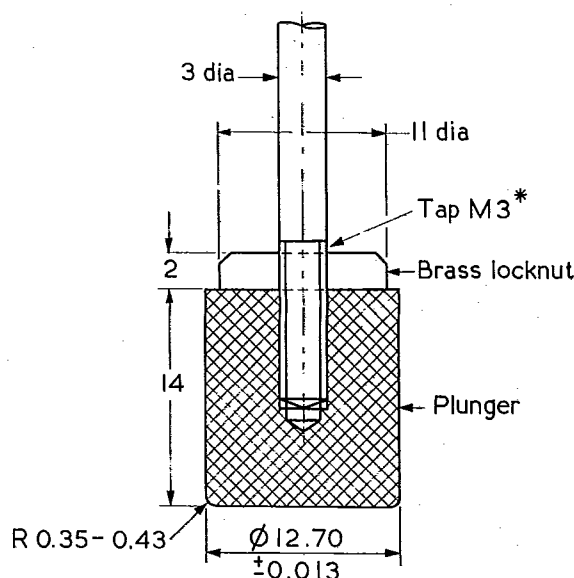
†In addition to the gel testing instrument described in clause 7, the following may be used for comparative testing:

(1) The Gelatine and Glue Research Association Jelly Tester, (Saunders & Ward, Proc. 2nd Rheological Congress 1954, Butterworth Ltd. London)

(2) Food Industry Research Association (FIRA) Jelly Tester (Campbell JSCI 1938, 57, 413)

For information as to the availability, apply to the Central Enquiries Section, British Standards Institution, enclosing a stamped, addressed envelope for a reply.

‡Water complying with the requirements of BS 3978 is suitable.



All dimensions are in millimetres.

*See BS 3643 : Part 2 for limits and tolerances for coarse pitch series threads and BS 1157 for tapping drill sizes.

NOTE 1. The diameter and radius given for the plunger are mandatory, whilst other dimensions are for guidance only.

NOTE 2. Some commercially available apparatus may not have plungers which conform to the above dimensions and radius of curvature and it is essential that this be ascertained particularly with reference to the radius of curvature.

Figure 1. Detail of plunger for gelometer

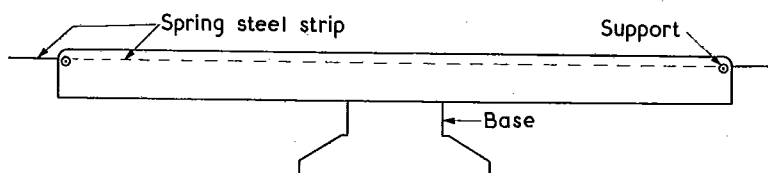


Figure 2. Dummy Bloom device

9. Methods for the determination of viscosity

9.1 Method 1. U-tube viscometer

9.1.1 Principle. The viscosity of a sample of the glue is determined at 60°C with a U-tube viscometer. (See also the foreword.)

9.1.2 Apparatus. The following apparatus is required.

9.1.2.1 Any type of glass capillary viscometer, as described in BS 188 is suitable. It is essential that the time of efflux is within the range of accuracy of the chosen instrument. The viscometer shall be calibrated as described in BS 188.

NOTE. For accurate work, it is recommended that the factor of the viscometer be determined by the National Physical Laboratory. The minimum time of flow for accurate work is usually 100 s. Other types of viscometers may be used, provided that they are accurately calibrated in SI units (mm^2/s) or in centistoke.

9.1.2.2 Thermostatic bath, capable of being maintained at $60 \pm 0.2^\circ\text{C}$.

9.1.3 Procedure. Place 15 g of the powdered sample (see clause 5) in a corked bottle or flask and add 105 ml of cold water, complying with the requirements of BS 3978.

Swell and dissolve the sample in the manner specified in 5.3. Pour the liquid through a funnel loosely plugged with cotton wool into the viscometer and allow to stand in the

thermostatic bath, maintained at $60 \pm 0.2^\circ\text{C}$, for 15 min before taking the measurement as described in BS 188.

NOTE. A sintered glass filter of porosity 2, as specified in BS 1752, may be used instead of the cotton wool plug.

9.1.4 Expression of results. Express the results either as the kinematic viscosity in $10^{-1} \text{ mm}^2/\text{s}$ (= 1 millistoke) or as the dynamic viscosity in 10^{-1} mPa s (= 1 millipoise).

NOTE. For most routine test purposes it is reasonable to use 1.02 g/ml as the density of the solution in calculating the dynamic viscosity from the kinematic viscosity.

9.1.5 Test report. The report shall state the following:

- a designation of the glue, giving all information required for complete identification of the sample;
- the result of the test together with full description of the apparatus used and any circumstances which may have affected the result;
- the method used i.e. method 1, clause 9 of BS 647 : 1981.

9.2 Method 2. American pipette

9.2.1 Introduction. This method is a convenient secondary method which can be used for production control. The method is suitable for the range of viscosities of 2.5 mPa s to 20 mPa s (25 mP to 200 mP).

1 cSt = $1 \text{ mm}^2/\text{s}$, 1 mPa s = 1 cP.