INTERNATIONAL STANDARD

ISO 9665

Second edition
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Adhesives — Animal glues — Methods of sampling and testing

Adhésifs — Colles d'origine animale — Méthodes d'échantillonnage et d'essai

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9665 was prepared by Technical Committee ISO/TC 61, *Plastics,* Subcommittee SC 11, *Products.*

This second edition cancels and replaces the first edition (ISO 9665:1993), annex A of which has been updated.

Annex A of this International Standard is for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

Printed in Switzerland

Introduction

The physical and chemical properties of animal glue depend firstly on the nature of the raw material and secondly on the methods of processing. It is not possible to develop 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.

Amongst these properties, the gel strength or viscosity, or both, are usually included as general indices of quality. The foam test is of interest when the glue is to be applied by special machines. Many of the physical tests (e.g. gel strength, water absorption, foam) are of an empirical character, but if the methods are carefully followed consistent results are obtainable which will provide useful information both to the manufacturer and user.

Small samples of glue rapidly change their moisture content in response to changes in atmospheric humidity. It is essential to keep the moisture content of samples unchanged after they are taken. The use of sealed waterproof storage containers is recommended for this purpose.

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 problem causing unnecessary disputes, it is recommended that, when gel 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.

Adhesives — Animal glues — Methods of sampling and testing

1 Scope

This International Standard specifies the methods to be used for sampling and testing bone and skin glues in the form of powder, granules, pearls or cubes.

NOTE 1 The methods may be extended to the testing of other forms of animal glues by suitable calculation of the equivalent dry glue content.

The precision of the test methods included in this International Standard is not known because interlaboratory data are not available. When interlaboratory data are obtained, precision statements will be added to the corresponding test methods at the next revision.

NOTE 2 For details of precision statements, refer to ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3105:1994, *Glass capillary kinematic viscometers — Specifications and operating instructions.*

ISO 4788:1980, *Laboratory glassware — Graduated measuring cylinders.*

3 Definitions

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

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

3.2 gel 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 General

The results of the analysis of glue carried out in accordance with this International Standard are limited in their practical use by the degree to which the 1 kg test sample represents the whole consignment.

4.2 Initial selection of sample

The containers shall be selected from various parts of the consignment in such a manner that the widest representation is obtained. The number of containers to be sampled shall be fixed by agreement between purchaser and vendor. State the number of containers sampled in the test report.

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. This sample is called the preliminary sample.

Take appropriately sized samples 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.

Store these preliminary samples in clean, dry, airtight, non-absorbent containers until required.

NOTE — For further guidance, reference should be made to ISO 3951:1989, *Sampling procedures and charts for inspection by variables, for percent nonconforming*, and ISO 8213:1986, *Chemical products for industrial use — Sampling techniques — Solid chemical products in the form of particles varying from powders to coarse lumps.*

4.3 Final selection of sample

Mix the preliminary samples thoroughly and take one or more samples from the bulk, each weighing not less than 1 kg, and store them in clean, dry, airtight, non-absorbent containers. These samples shall be known as the laboratory samples.

5 Preparation of test sample

5.1 Reduction of solid (powdered, pearl, cube and granulated) glue sample

Grind the sample by hand in a mortar, or alternatively in a laboratory disintegrator, of a type capable of grinding the sample to a particle size of 3 mm or smaller. This sample is called the test sample. 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 samples so obtained in two air-tight containers, one container for the test sample for moisture content (see clause 6) and the second container for all other tests.

The type of disintegrator used to grind the sample shall be agreed on between the purchaser and vendor because different types of disintegrator generate different amounts of heat and have different effects on the moisture content of the sample.

Because of the loss of moisture in grinding, take a separate sample for the moisture content test from the material obtained by the preliminary breaking up, before the material is put through the disintegrator. This small separate sample is then powdered by hand in a mortar and pestle, or cut with scissors, and is suitable for the moisture content test. Make adjustment for the moisture content of the mechanically ground sample by comparison with the hand-ground sample, which is considered 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 is determined by calculation.

Place 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 will result in a decrease of about 2,5 % in gel 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. Weigh the specified quantity of the powdered sample for each test separately, rather than by working from a large quantity of stock solution.

Table 1 — Glue concentrations

Clause	Test	Mass of glue g	Volume of water ml
6	Moisture content	1	10
7	Gel strength (see 7.5.2)	15 (7,5)	105 (105)
8	Comparison of gel strength	5 to 10	50
9	Viscosity	15	105
10	Softening point	37,5	75
11	Setting point	37,5	75
12	Foam	5	50
13	pH	1	100
14	Grease	10	15
16	Keeping quality	20	80

5.3 Dissolving solid glues

Weigh the test portion in a beaker and add the requisite amount of cold distilled water, stirring with a thin metal or glass rod. Place a watch glass over the mouth of the beaker and allow the sample to soak for 2 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 the 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 test portion has all dissolved, remove the beaker from the water bath.

If the sample gives a gel strength above 400 g Bloom, prepare the gel at a concentration of 6,67 %, and note this observation in the report. The results on a 6,67 % solution are sometimes expressed as “single Bloom”, and those on a 12,5 % solution as “double Bloom”.

6 Determination of moisture content

6.1 Principle

A weighed test portion of the glue is maintained at 105 °C for 18 h and is then reweighed.

6.2 Apparatus

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, capable of being maintained at 105 °C ± 1 °C.

6.2.3 Water bath.

6.2.4 Balance, capable of weighing to the nearest 0,01 g.

6.2.5 Desiccator.

6.3 Procedure

Weigh into the tared dish (6.2.1), to the nearest 0,01 g, about 1 g of the test sample for moisture content (see 5.1, third paragraph).

Add 10 ml of distilled water and allow the glue to soak. Place the dish on a warm water bath (6.2.3) 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 (6.2.2) set at 105 °C ± 1 °C, and allow it to remain there for 18 h ± 1 h, during which time the oven door shall not be opened. Remove the dish from the oven and, after allowing it to cool in a desiccator (6.2.5), weigh it.

Carry out the determination in duplicate.

6.4 Expression of results

Calculate the moisture content (M) as a percentage by mass, using the equation:

$$M = \frac{m_0 - m_1}{m_0} \times 100$$

where

m_0 is the initial mass, in grams, of the test portion;

m_1 is the mass, in grams, of the test portion after drying.

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

6.5 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for the complete identification of the glue tested;

c) the number of containers sampled and the mass of glue taken from each container;

d) the results of the test, including the individual values, and any circumstances that may have affected the results.

6.6 Variation of gel strength and of viscosity with moisture content

If greater precision is required, adjust the result to allow for variations in the moisture content.

An approximate formula for the change in gel strength (ΔF) as a result of a change in moisture content (ΔM) is:

$$\Delta F = \frac{-2F_1 \times \Delta M}{100 - M_1}$$

where F_1 is the gel strength at a percentage moisture content M_1.

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

NOTE — If M_1 is taken as 15 %, then $\Delta F = F/42{,}5$ for each 1 % change in moisture content. As an example of the scale of this effect, an increase in moisture content from 15 % to 16 % reduces the measured gel strength of a 250 g gel strength glue by 6 g.

There is no satisfactory formula for the change of viscosity, $\Delta\eta$, with moisture content, ΔM, but a rough guide is given by

$$\Delta\eta = -2{,}6\eta_1 \times \frac{\Delta M}{100}$$

This formula applies to 12,5 % solutions of glue.

7 Measurement of gel strength

7.1 Principle

The gel strength of the sample for the test is measured under arbitrary conditions. A suitable instrument measures the force necessary to give a 4 mm depression in a gel of 12,5 % concentration by mass, matured for 16 h to 18 h at 10 °C, using a standard plunger (7.2.4).

By convention, this force has normally been expressed as a mass in grams (see, however, 7.6).

7.2 Apparatus

7.2.1 Wide-neck bottle, of internal diameter 59 mm ± 1 mm, approximately 85 mm high, having a capacity of approximately 155 ml. The bottle shall have a stopper approximately 43 mm in diameter, pierced centrally with an air vent about 2,5 mm in diameter.

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

7.2.3 Totally enclosed thermostatically controlled bath, fitted with a thermometer, and capable of being maintained at 10 °C ± 0,1 °C.

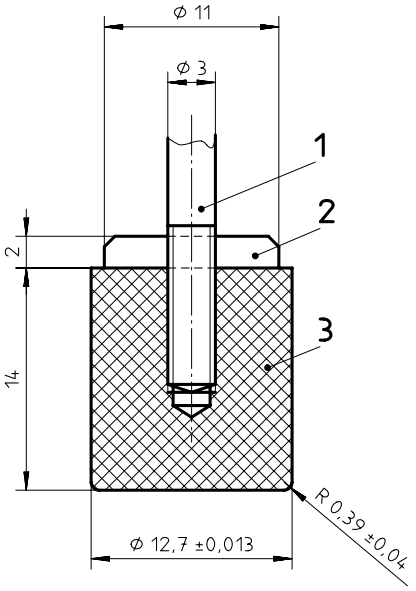
7.2.4 Plunger, made of a suitable, stable polymer with a diameter of 12,7 mm and an edge radius of 0,39 mm ± 0,04 mm (see figure 1).

NOTES

1 The diameter of the plunger and its edge radius are mandatory; other dimensions are for guidance only.

2 Some types of commercially available apparatus may have plungers that do not conform to the dimensions specified for the diameter and edge radius, and it is essential that compliance, particularly with reference to the radius of curvature, be ascertained.

Dimensions in millimetres



Key

1 Tap
2 Brass locknut
3 Plunger

Figure 1 — Detail of plunger for gelometer

7.2.5 Gel-testing instrument (penetrometer), 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 mm ± 0,01 mm, either at a constant rate of loading not exceeding 40 g/s or at a constant rate of penetration not exceeding 0,8 mm/s. (See annex A for details of suitable instruments.)

7.2.6 Balance, capable of weighing to the nearest 0,01 g.

7.3 Dissolving the sample

Weigh out, to the nearest 0,01 g, 15 g of the test sample (see clause 5), and dissolve this test portion 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 °C ± 1 °C. Particular care is necessary to see that the glue is all dissolved and that the solution is homogeneous. Place a finger over the hole in the stopper and invert the bottle several 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

7.5 Procedure for determining gel strength

NOTE — See annex 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 penetrometer 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. If the gel strength is above 400 g, repeat the test using a solution prepared by dissolving 7,5 g of the test sample in 105 ml of water to give a solution concentration of 6,67 %.

7.6 Expression of results

Express the result as the force, in newtons, required to depress the plunger 4 mm into the gel.

NOTE — In some instruments, the force is applied by the addition of weights to a container attached to a counter-balanced plunger, and it has been customary with such instruments to quote the gel strength as “grams Bloom”, this being numerically equal to the mass of the container plus the added weights. To enable comparison of current results with earlier records, it should be noted that 1 N is numerically equal to 101,972 “grams Bloom”.

7.7 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for the complete identification of the glue tested;

c) the number of containers sampled and the mass of glue taken from each container;

d) the result of the test, the concentration of the glue solution and any circumstances which may have affected the results;

e) the method and instrument used.

7.8 Validation of the test method

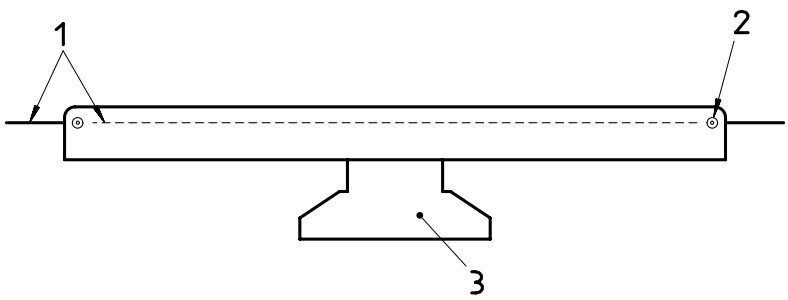
The following three criteria shall be satisfied in order to validate the test method:

a) The diameter of the gelometer plunger and the radius of curvature of its lower circular edge shall conform to the dimensions given in figure 1.

NOTE 1 A profilometer is a satisfactory instrument for measuring the radius of curvature.

b) The gelometer used shall be calibrated using a device consisting of a strip of metal, supported near each end, which offers a pre-determined resistance, simulating that of the surface of a gel being tested.

NOTE 2 This device is sometimes known as a “dummy Bloom” device and can usually be obtained from the manufacturer of the test equipment. (See figure 2.)



Key

1 Spring steel strip

2 Support

3 Base

Figure 2 — Dummy Bloom device