भारतीय मानक

Indian Standard

IS 11022 : 2024
ISO 9665 : 1998

गोंद, हड्डी, त्वचा/मांस और मछली के गोंद के नमूने एवं परीक्षण पद्धतियाँ

(पहला पुनरीक्षण)

Methods of Sampling and Test for Glues, Bone, Skin/Fleshings and Fish Glues

(First Revision)

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Methods of Sampling and Test for Plastics Sectional Committee, PCD 27

NATIONAL FOREWARD

This Indian Standard (First Revision) which is identical to ISO 9665 : 1998 ‘Adhesives — Animal glues — Methods of sampling and testing’ issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendations of the Methods of Sampling and Test for Plastics Sectional Committee and approval of the Petroleum, Coals and Related Products Division Council.

This standard was first published in 1984. This revision has been undertaken to align the standard with the ISO 9665 : 1998.

The text of ISO standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words ‘International Standard’ appear referring to this standard, they should be read as ‘Indian Standard’; and

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to the following International Standard for which Indian Standard also exists. The corresponding Indian Standard that is to be substituted in its place is listed below along with its degree of equivalence for the edition indicated.

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 4788 : 1980 Laboratory glassware — Graduated measuring cylinders	IS 878 : 2008/ISO 4788 : 2005 Laboratory glassware — Graduated measuring cylinders (*second revision*)	Identical

The Committee has reviewed the provisions of the following International Standards referred in this adopted standard and has decided that it is acceptable for use in conjunction with this standard:

International Standard	*Title*
ISO 3105 : 1994	Glass capillary kinematic viscometers — Specifications and operating instructions

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

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.

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Indian Standard

METHODS OF SAMPLING AND TEST FOR GLUES, BONE, SKIN/FLESHINGS AND FISH GLUES

(First Revision)

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.

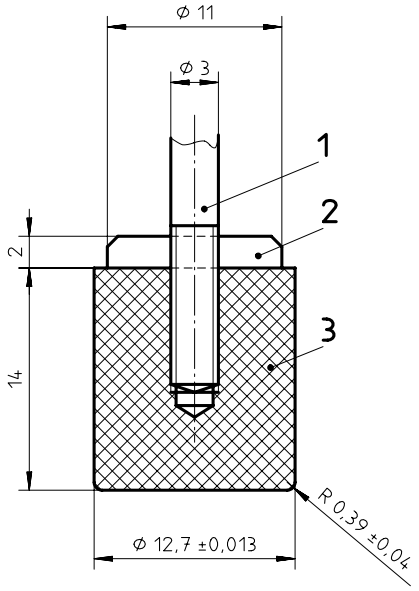
7.2.4 Plunger, made of a suitable, stable polymer with a diameter of 12,7 mm and an edge radius of 0,39 mm ± 0,04 mm (see figure 1).

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

To prevent cracking, allow the bottle to cool for 15 min at room temperature, and then place in the enclosed thermostatic bath (7.2.3), maintained at 10 °C ± 0,1 °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 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

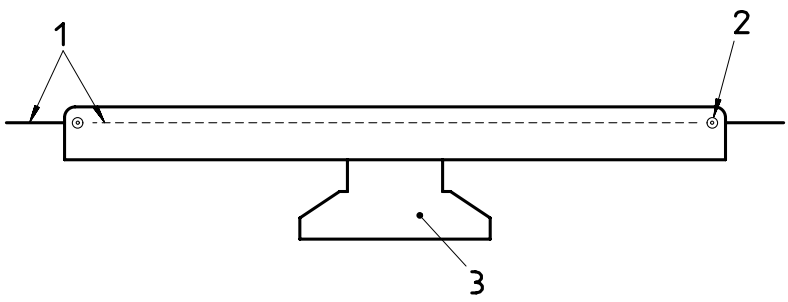
The following three criteria shall be satisfied in order to validate the test method:

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

c) The full procedure shall be checked using samples standardized against reference samples of known gel strength to ensure that differences in sample preparation and operating technique have not introduced variations.

All standard samples shall be corrected for variations due to moisture content if the moisture content differs by more than 0,4 % from the value at the time of standardization (see 6.6).

8 Comparison of gel strength

8.1 Principle

Solutions of glues to be compared are prepared under identical conditions, allowed to gel for 16 h and their gel strengths compared using any suitable instrument.

NOTE — This method is frequently used to compare batches of glues and to compare a given sample of glue against a reference sample.

8.2 Apparatus

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

8.2.2 Thermostatically controlled bath, capable of being maintained to within 0,1 °C at a temperature between 5 °C and 30 °C (for use if numerical results are required).

8.2.3 Beaker, glass, 150 ml capacity.

8.2.4 Instrument for comparing gel strengths.

8.3 Procedure

Weigh, to the nearest 0,01 g, 5 g to 10 g, according to grade and use, of each test sample (see clause 5) into a 150 ml beaker (8.2.3), and add 50 ml of cold distilled water from a pipette. Dissolve the test portion 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.

Cool the container for 16 h using method a) if numerical results are to be obtained, or either method a) or method b) if only comparative (ranking) results are required:

a) place in a water bath (8.2.2), thermostatically controlled at a temperature to be stated in the test report;

b) place in a bath of cold running water.

Numerical values obtained using methods a) and b) above are to be used only for ranking samples of glue and not for obtaining values to be quoted as “gel strengths”, for which the method given in clause 7 is used.

8.4 Expression of results

Express the results by identifying the glue samples and listing them in the order of their apparent gel strengths.

If the temperature of gelling was controlled, this temperature shall be stated, and the numerical values of the apparent gel strengths as indicated by the instrument (8.2.4) selected to measure them may be quoted as “comparative strengths”.

8.5 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 concentrations of the glue solutions used and, if numerical results are to be quoted, the gelling temperature, in degrees Celsius;

e) the results of the test, the instrument used and any circumstances that may have affected the results.

9 Determination of viscosity

9.1 Method 1: Glass-capillary viscometer

9.1.1 Principle

The viscosity of a sample of the glue is determined at 60 °C with a glass-capillary viscometer. (See also the Introduction.)

9.1.2 Apparatus

9.1.2.1 Glass-capillary viscometer, of any type as described in ISO 3105. It is essential that the time of efflux is within the range of accuracy of the chosen instrument. The viscometer shall be calibrated.

9.1.2.2 Thermostatically controlled bath, capable of being maintained at 60 °C ± 0,2 °C.

9.1.3 Procedure

Weigh, to the nearest 0,01 g, 15 g of the test sample (see clause 5) into a corked bottle or flask and add 105 ml of cold distilled water. Swell and dissolve the test portion in the manner specified in 5.3. Pour the liquid through a funnel loosely plugged with cotton wool (see however the note) into the viscometer (9.1.2.1) and allow to stand in the thermostatically controlled bath (9.1.2.2), maintained at 60 °C ± 0,2 °C, for 15 min before making the measurement.

NOTE — A sintered-glass filter of suitable porosity may be used instead of the cotton-wool plug.

9.1.4 Expression of results

Express the result either as the kinematic viscosity, in square millimetres per second (1 mm^2/s = 1 cSt), or as the dynamic viscosity, in millipascal seconds (1 mPa·s = 1 cP).

NOTES

1 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.

2 The present industry convention is to express viscosity in millipoises (1 cP = 10 mP).

9.1.5 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 result of the test, together with a full description of the apparatus used and any circumstances which may have affected the results;

e) the method used, i.e. method 1 of this International Standard.

9.2 Method 2: American pipette

9.2.1 General

This method is a convenient secondary method which may be used for production control. The method is suitable for the range of viscosities of 2,5 mPa·s (2,5 cP) to 20 mPa·s(20 cP).

9.2.2 Principle

The viscosity of a sample is determined at 60 °C by measuring the flow time of the sample from a standard pipette.

9.2.3 Apparatus

9.2.3.1 American pipette and **container** (see figure 3). This consists of a modified 100 ml pipette immersed in a thermostatic bath which may be in the form of an inverted bell jar or transparent plastic container, and which is capable of being maintained at 60 °C ± 0,2 °C. The bath shall have a means for circulating water.

NOTES

1 A suitable method for circulating the water is to use a small external air pump which produces a fine stream of air bubbles.

2 Any other 100 ml pipette with a calibrated precision tube may be used.

9.2.3.2 Timing device.

9.2.3.3 Thermostatically controlled bath, capable of being maintained at 65 °C ± 2 °C.

9.2.4 Procedure

Prepare the samples as described in 9.1.3, but place the bottle or flasks in a thermostatically controlled bath (9.2.3.3) at a temperature of 65 °C ± 2 °C for a period of not more than 15 min. Close the lower end of the pipette with the finger and pour sample liquid into the pipette in the thermostatic bath (see 9.2.3.1), maintained at 60 °C ± 0,2 °C, as quickly as possible, without trapping air, until the level of the solution is approximately 1 cm above the upper mark. Place the bottle or flask below the end of the pipette. Insert the thermometer, which has been maintained at 60 °C to 65 °C in warm water, into the bath and move slowly up and down until a temperature of 60 °C is shown when the thermometer bulb is midway between the top and bottom of the bulb of the pipette. Remove the thermometer and adjust the level of the solution in the pipette so that the bottom of the meniscus coincides with the upper mark on the pipette.

Remove the finger from the end of the pipette and measure the time for the pipette to deliver 100 ml of the solution.

Clean the interior of the apparatus immediately after the determination by rinsing with water at 60 °C.

9.2.5 Calibration of the pipette

Calibrate the pipette by one of the following procedures:

a) carry out measurements using standard-viscosity oils and plot flow time against viscosity;

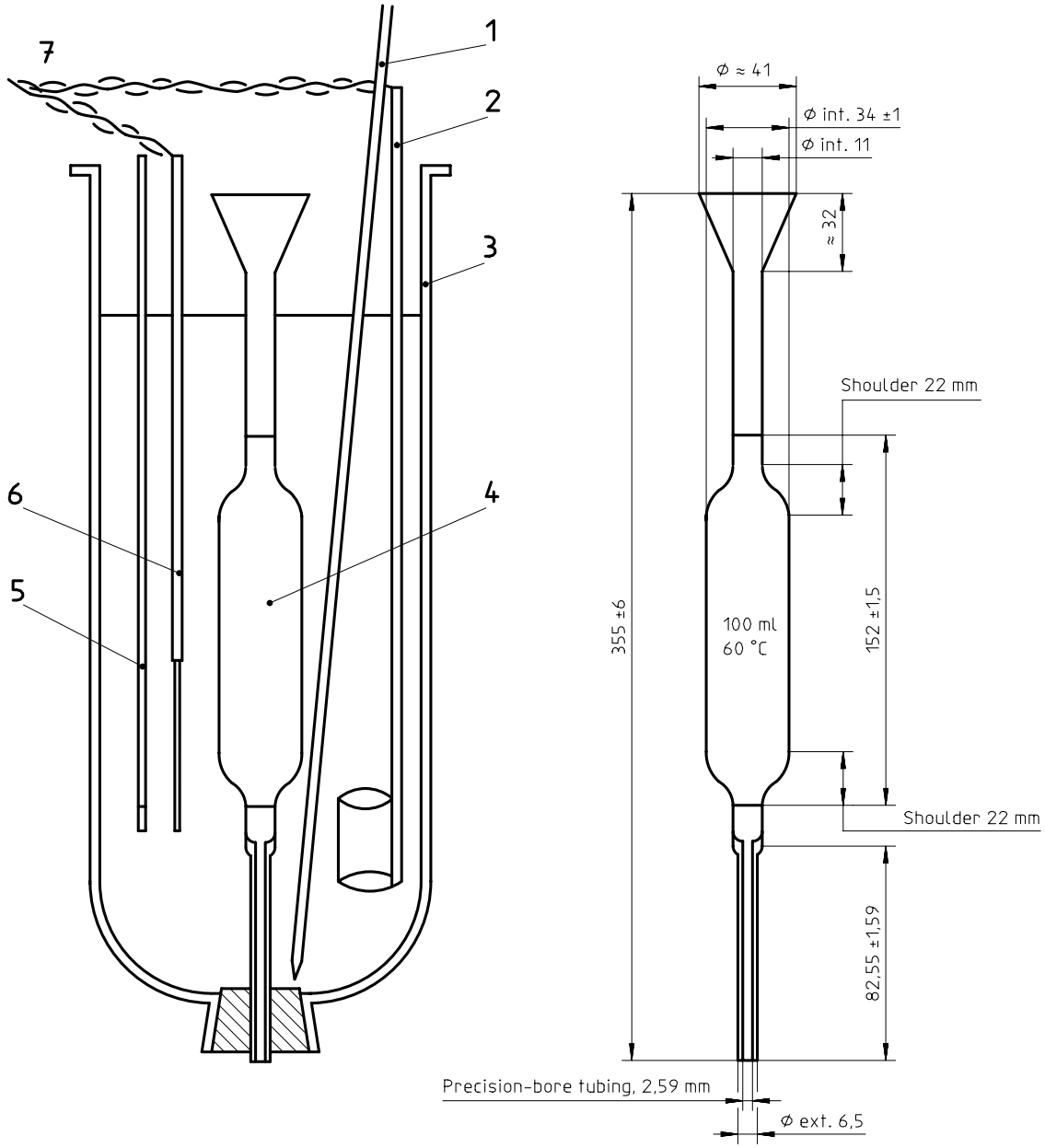
b) compare results obtained with the pipette with results obtained with viscosities specified in method 1 and plot flow time against viscosity.

9.2.6 Expression of results

Express the results either as the kinematic viscosity, in square millimetres per second (1 mm^2/s = 1 cSt), or as the dynamic viscosity, in millipascal seconds (1 mPa·s = 1 cP), at 12,5 % concentration at 60 °C.

NOTE — The dynamic viscosity (measured in millipascal seconds or centipoises) is obtained by multiplying the kinematic viscosity (measured in square millimetres per second or centistokes) by the density of the glue solution at 60 °C, which may be taken as 1,02 g/ml.

Dimensions in millimetres



Key

1 Air tube
2 Heater
3 Bell jar
4 American pipette
5 Thermometer
6 Thermostat
7 To relay

Figure 3 — American pipette and suitable container

9.2.7 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 and any circumstances which may have affected the result;

e) the method used, i.e. method 2 of this International Standard.

10 Determination of softening point

10.1 General

This method gives an arbitrary but reproducible softening point for gels in which the transition is not very sharp.

10.2 Principle

A cup containing glue gel of 33,3 % concentration is suspended by a glass rod held in the gel in a beaker of water which is heated slowly until the cup falls from the rod.

10.3 Apparatus

10.3.1 Brass cup, 22 mm in height and 17 mm in external diameter at the bottom. Its wall thickness is such that the mass is 7,0 g.

10.3.2 Glass rod, 40 mm long and 3 mm in diameter, flattened at one end to a disc of 9 mm diameter, and fashioned at the other end into a hook.

10.3.3 Thermostatically controlled bath, capable of being maintained at 10 °C ± 0,1 °C.

10.3.4 Water bath.

10.3.5 Thermometer, graduated to 0,05 °C.

10.4 Procedure

Weigh, to the nearest 0,01 g, 37,5 g of the test sample (see clause 5) into a beaker and add 75 ml of cold distilled water. Swell and dissolve this test portion in the manner specified in 5.3. Allow entrained air to escape and pour the glue solution into the cup (10.3.1). Insert the glass rod (10.3.2) into the gel to the base of the cup and maintain it in an upright position for 16 h in the thermostatic bath (10.3.3), maintained at 10 °C ± 0,1 °C, for gelation to occur. Totally immerse the cup and suspend it by means of the glass rod in a beaker of water at 15 °C. Place the beaker in the water bath (10.3.4) at 20 °C and heat the water in the latter so that the temperature of the water in the beaker rises at the rate of 0,25 °C in 60 s ± 10 s. Note the temperature of the water in the beaker when the cup falls from the rod.

10.5 Expression of results

Express the softening point of the gel as the temperature of the water in the beaker at which the cup fell from the rod.

10.6 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 and any circumstances which may have affected the result.

11 Determination of setting point

11.1 Principle

A 33,3 % solution of glue is cooled from 45 °C to 20 °C, stirring by means of a thermometer. The thermometer is pulled out at regular intervals and the temperature at which the thread of glue solution picked up with the thermometer first breaks is recorded as the setting point for the glue.

11.2 Apparatus

11.2.1 Beaker, 150 ml, tall-form, of borosilicate glass.

11.2.2 Thermostatically controlled bath, capable of being maintained at 20 °C ± 0,5 °C.

11.2.3 Thermometer, graduated to 0,1 °C.

11.3 Procedure

Weigh, to the nearest 0,01 g, 37,5 g of the test sample (see clause 5) into the beaker (11.2.1) and add 75 ml of cold distilled water. Swell and dissolve this test portion in the manner specified in 5.3. Adjust the temperature of the solution to 45 °C.

Place the beaker and contents in the thermostatically controlled bath (11.2.2), maintained at 20 °C ± 0,5 °C, and stir the glue solution regularly with the thermometer (11.2.3). Pull the thermometer out periodically and note the highest temperature at which the thread of glue picked up with the thermometer first breaks.

11.4 Expression of results

Express the setting point of the glue as the highest temperature at which the thread of glue picked up with the thermometer just breads as the solution cools.

11.5 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 result of the test and any circumstances which may have affected the result.

12 Determination of foam

12.1 General

This test is intended to assist the assessment of the foaming tendency of a given sample. However, it is not possible to simulate in a simple laboratory test all the conditions that may arise in the actual use of the glue. The results should therefore be applied with caution.

12.2 Principle

The volume of foam and the rate of collapse of the foam produced by shaking a solution of the glue are determined.

12.3 Apparatus

12.3.1 Stoppered glass measuring cylinder, 100 ml capacity, complying with the requirements of ISO 4788, and in which the air space above the graduations corresponds to 50 ml ± 2 ml.

12.3.2 Water bath, at least 250 mm in diameter and 200 mm high, the water coming up to the 70 ml mark on the cylinder. The bath shall be capable of being maintained at a temperature of 45 °C ± 1 °C.

12.4 Procedure

Weigh, to the nearest 0,01 g, 5 g of the test sample (see clause 5) into the dry cylinder (12.3.1) and add cold distilled water to just below the 50 ml mark. Swell and dissolve this test portion in the manner specified in 5.3 and adjust the volume to 50 ml at 45 °C ± 1 °C. Place the cylinder containing the glue solution in the water bath (12.3.2) at 45 °C ± 1 °C, for 30 min. At the end of this period, shake the cylinder vigorously with a throw of about 300 mm at a rate of three shakes per second for 1 min. Note the volume of the foam above the 50 ml mark. Loosen the stopper and then place the cylinder immediately in the water bath at 45 °C ± 1 °C and allow it to remain there until the height of the solution corresponds to 45 ml. Note the time taken for the solution/foam interface to reach the 45 ml mark, together with the volume of foam. Note also the time for the foam to disappear completely. With some glue solutions, the solution/foam interface does not reach the 45 ml mark and therefore the above instructions cannot be followed. In such cases, it is usual for the interface to fall just below the 50 ml mark and for the level to drop a further 1 ml to 2 ml due to the further collapse of liquid foam and then to rise slightly owing to the further collapse of aerial froth. In such cases, read the volume at the lowest level and report the volume of froth at that time, e.g. 10 ml in 1 min, making a note in the test report that the glue solution did not reach the 45 ml mark.

It is important that this determination be carried out in a place free from draughts.

NOTE — A commercial shaking device capable of following the above procedure is convenient to use.

12.5 Expression of results

Express the results of the test as follows:

a) the initial volume of foam above the 50 ml mark, in millilitres;

b) the time, in minutes, for the solution/foam interface to reach the 45 ml graduation and the corresponding volume of foam, in millimetres;

c) the time, in minutes, for the foam to disappear completely (if this time is over 90 min, report as “over 90 min”).

12.6 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 and any circumstances which may have affected the results.

13 Determination of pH

13.1 Principle

The pH of a 10 g/l solution is determined electrometrically or, for approximate work, colorimetrically.

13.2 Procedure

Weigh out, to the nearest 0,01 g, 1 g of the test sample (see clause 5) and dissolve this test portion by shaking after careful addition to a small quantity of warm, recently boiled, distilled water in a stoppered flask of chemically resistant glass. Then dilute to 100 ml with the same water. After swirling and allowing to cool to room temperature, determine the pH value of the solution by actual measurement of the potential by a recognized method. Care shall be taken throughout the operation to minimize absorption of carbon dioxide from the air.

For approximate work, colorimetric methods may be used.

13.3 Expression of results

Record the potential measured by the instrument in units of pH.

13.4 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 and any circumstances which may have affected the result.

14 Determination of grease content

14.1 Principle

After saponification of the sample with alcoholic potassium hydroxide solution, the ether-soluble material is recovered and weighed.

14.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

14.2.1 Acetone.

14.2.2 Diethyl ether.

14.2.3 Potassium hydroxide solution in ethanol, c(KOH) = 1,0 mol/l.

14.2.4 Hydrochloric acid, 100 g/kg solution, c(HCl) = 3 mol/l.

14.3 Procedure

Weigh out, to the nearest 0,01 g, 10 g of the test sample (see clause 5) and soak this test portion in 15 ml of cold water in a porcelain dish until the water is all absorbed. Then heat the dish on a water bath until dissolution is

complete. Add 40 ml of the potassium hydroxide solution (14.2.3). Evaporate the solution on a water bath and dry the residue in an oven at 105 °C. Dissolve the residue in 100 ml of the hydrochloric acid solution (14.2.4) and keep on a steam bath for 30 min. Transfer the solution to a separating funnel and dilute to approximately 100 ml with water.

The following procedure shall be carried out in a fume cupboard.

When cool, add 50 ml of the ether (14.2.2) and mix thoroughly, but not so vigorously as to form a stable emulsion. Allow to stand until completely separated and run the ethereal solution into another separating funnel. Repeat this twice with further 50 ml portions of ether. Wash the combined ethereal solution with 50 ml portions of cold water until free from acid (three or four washings). Transfer to a tared flask and distil off the ether in a steam bath, then add 3 ml of the acetone (14.2.1) and distil this off for 2 min on the steam bath in a gentle current of air. When dry, cool in a desiccator and weigh to the nearest 0,01 g.

14.4 Expression of results

Calculate the grease content, expressed as a percentage by mass of the sample, using the formula:

$$\frac{m_1}{m_0} \times 100$$

where

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

m_1 is the mass, in grams, of the dried ether extract of the hydrolysate.

14.5 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 result of the test and any circumstances which may have affected the result.

15 Determination of ash

15.1 Principle

A powdered test portion is ashed carefully at a temperature not exceeding 500 °C to avoid loss of chlorides. The mass of the ash is then determined.

15.2 Apparatus

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

15.2.2 Platinum or silica dish.

15.3 Procedure

Ash carefully about 5 g, weighed to the nearest 0,01 g, of the test sample (see clause 5) in a tared platinum or silica dish. Use a moderate temperature (e.g. over a low flame or in a muffle furnace) until all the organic matter has been charred, and complete the ashing by raising the temperature to 495 °C ± 5 °C until constant weight is obtained.

If there is any difficulty in obtaining a carbon-free ash, the following procedure may be adopted. Ash till black, extract with water, ash the carbon, add the water extract, and evaporate to dryness.

Take particular care not to overheat. After charring, the carbonized mass may be pressed down onto the bottom of the dish by a glass rod or agate pestle.

15.4 Expression of results

Calculate the amount of ash, expressed as a percentage by mass, using the formula:

$$\frac{m_1}{m_0} \times 100$$

where

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

m_1 is the mass, in grams, of the ash.

15.5 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 result of the test and any circumstances which may have affected the result.

16 Determination of keeping quality

WARNING — In drafting this method, a level of microbiological expertise in the reader has been assumed. Readers less familiar with microbiological techniques are advised to inform themselves of the safeguards necessary for staff employed on microbiological work.

Particular attention is drawn to the possibility of the growth of pathogenic organisms, which requires that special care be exercised when carrying out this method and when disposing of the waste material.

16.1 General

The rate of growth of fungi, yeasts and bacteria and the loss of viscosity and gel strength are dependent, amongst other factors, on temperature, water content and preservative concentration. A dry glue is stable in storage for many years. Once a solid glue is dissolved in water or a liquid glue is diluted, more water is available for growth, and the concentration of preservative (if any) is decreased. Therefore any practical test of keeping quality should preferably be made under the intended conditions of storage and use.

The test is intended simply as an indicator of the behaviour of the glue in practice, and is not to be taken as a substitute for a more complete bacteriological examination.

16.2 Principle

Evidence of microbiological activity is sought daily in a 20 % solution of the glue, prepared under normal non-sterile conditions and maintained at 37 °C.

16.3 Procedure

For a general test, prepare a 20 % solution of the test sample (see clause 5) in distilled water by the method specified in 5.3. Allow the solution to cool to room temperature (or to just above its gel point, whichever is the higher) and transfer it to a suitable set of clear glass jars or conical flasks. Half fill each container and cover its mouth with polyethylene film to prevent drying of the test portion. Any spatulas, beakers, etc., used in the transfer shall be clean and dry but do not necessarily have to be sterile.

Store the test portions in an incubator maintained at 37 °C. Remove them daily from the incubator and examine immediately for putrefaction. Cool to room temperature or to below the normal gel temperature, whichever is the lower, and examine for liquefaction or mould growth. Replace the test portions as soon as possible in the incubator for a further day.

If required, the test can be extended by maintaining a similar set of test portions at a temperature of 23 °C ± 2 °C, which conditions may be more favourable for growth of some organisms.

Note the times when liquefaction, putrefaction, mould growth or any combination of these occurs.

16.4 Expression of results

Express the result as the time when liquefaction, putrefaction, mould growth or any combination of these occurs.

16.5 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 result of the test and any circumstances which may have affected the result.

Annex A
(informative)

Instruments for determining gel strength

A.1 General

Any mechanical or electromechanical test apparatus may be used which is able to press the standard plunger (see figure 1) into a prepared gel sample at the required rate and to measure the force required to do so. Currently available test apparatus which may be used for testing gels under a range of conditions is described in this annex.

A.2 Texture analyser gelometer[1)]

A.2.1 Principle

Detailed drawings are given in figure A.1, which shows the control panel and general details of the internal adjustments. The apparatus is able to vary both the depth and the rate of penetration of the standard plunger (see 7.2.4 and figure 1).

A.2.2 Test procedure

Place the test portion (prepared in accordance with 8.3) on the equipment and adjust it to a convenient height so that the probe is at least 10 mm from the surface of the test portion.

Start the test. The probe will then descend at a pre-set slow speed until a force of 4 g to 5 g is encountered: this is the “trigger point”. From this point, the probe begins the selected linear travel at the selected test speed. Upon making contact with the surface of the test portion, the probe will travel a further 4 mm into the sample. The reading on the digital display is the gel strength in grams Bloom.

A.3 Materials evaluation apparatus[1)]

This general-purpose low-force test instrument may be adapted for measurement of Bloom strength, with measurements linked to a chart recorder, printer or computer with suitable software. An example is given in figure A.2. A console controller is also available.

Test parameters may be pre-selected using the computer keyboard. Results are displayed in tabular form, with mean and standard deviation monitored in real time, as a batch is tested. Test results may be stored in a database or printed in detail for future reference.

A.4 Texture analyser[2)]

This instrument is a microprocessor-controlled texture analysis system which can be interfaced to a wide range of peripherals, including personal computers. It consists of two separate modules: the test bed, with a probe carrier which contains a very sensitive load cell, and the control console (keyboard). The test bed and control console are linked by cable to each other and to a computer. This analyser, with accessories, can be used for many different measurements involving accurate determinations of force, distance and time. Forces may be measured against set distances and distances may be measured to achieve set forces, with or without time delays. Results may be read directly from the keyboard or transmitted directly to a printer or computer. Alternatively, data may be passed to a chart recorder.

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1) A suitable apparatus is available from Leonard Farrell Ltd, North Mimms, Hatfield AL9 7SR, UK, Tel: +44 1707 264 488.

2) A suitable apparatus is available from Stable Micro Systems Ltd, Vienna Court, Lammas Road, Godalming, Surrey GU7 1JG, UK, Tel: +44 1483 427 345, Fax: +44 1483 427 600.

It includes three units:

a) The machine base unit (figure A.3) with all the mechanical parts of the system, including the gel-measuring probe, digital motors (step and up/down) together with the electronics required to interface with the load cell and drive the motor. The stepping motor, attached to a fine lead screw which winds the probe carrier up and down, moves one step every time a pulse is received from the microprocessor. Four hundred steps are required for each revolution of the lead screw, which moves the probe carrier by 1 mm. The system has an internal resolution of 0,002 5 mm. The pulses of the microprocessor are controlled by a quartz crystal clock, with a ratio of over 700:1 between the frequency of the master clock and the frequency of the clock motor drive. The distance of movement of the probe carrier is measured by counting the number of pulses sent to the stepping motor.

   When the required distance is reached (which is 4 mm for the measurement of Bloom strength, as required in this International standard), an interrupt signal is sent to the microprocessor so that it responds to the next operation, recording the measurement.

b) A keyboard console (figure A.4), containing the electronic items required to control machine movement and acquire the data for the display and printout.

c) A computer and dimension card, which includes the computer software providing the means for acquiring data and displaying it in real time. This acquired data may be stored, received or manipulated for detailed investigation.

These three units are connected by the following cables:

— A cable to carry signals between the machine base unit and the keyboard console. Power to the keyboard is also carried by this cable.

— A cable to carry the analogue load-cell signal, together with the digital motor signals (step and up/down) and other digital signals. This link is solely from the load cell to the computer, with no signals in the reverse direction.

— An RS232-type cable carrying information between the keyboard console and the computer. This link does not carry time-critical information; when no computer is used, the link can be used to carry signals to operate a serial printer (with different cable terminals).

— A “stop” cable, running between points in the base unit and the load cell to upper and lower mechanical limit stops.

When the texture analyser is switched on, all displays and lights are illuminated (typically for 2 s). Following this, the selected programme number is shown on the keyboard display. Any set-up changes are also shown. After a further 4 s, the system is ready for use.

On typical equipment as shown in the figures in this annex, there are three displays along the top of the console keyboard and four rows of lamps running downwards, a numerical keypad and sets of light- and dark-coloured operation keys. There is also a security key for protection against unauthorized alteration of settings and test programme parameters, so that these can be arranged permanently and only changed by the operator. When in the “test set-up” mode, the displays are used to show

— the probe speed during the test (in 0,1 mm/s steps);

— the test distance (in 0,1 mm steps);

— the test force, or count, or time (as indicated by the illuminated light on the right of the display).

It is possible to carry out three types of calibration on the texture analyser:

— the load cell;

— the probe height;

— the chart recorder (if fitted).

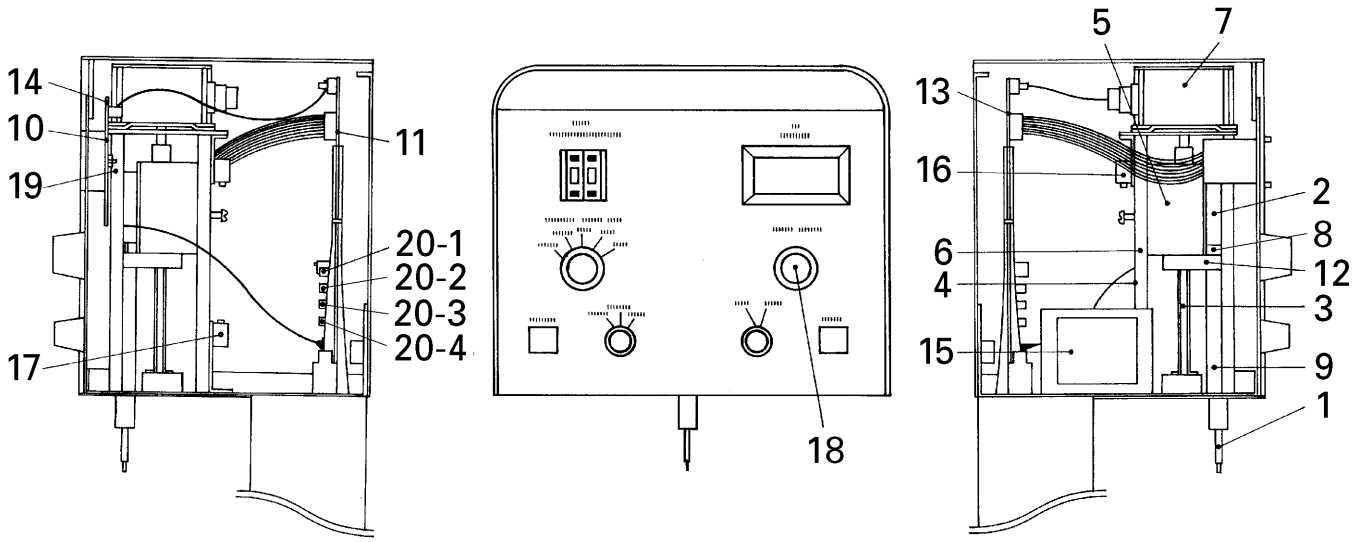
Calibration of the load cell is necessary for force measurement. This is carried out in the “test set-up” mode by placing a known weight on the circular pan on top of the load carrier, and the display reading checked with the known value, to ensure that it is within the force accuracy of 0,025 %.

The probe height is calibrated in the “machine configure” mode by attaching the probes to the probe carrier and base as required. In the “fast” mode, with the calibrate button pressed, the probe moves towards the base at 1 mm/s. When the texture analyser detects the trigger force, it stops the probe carrier and zeroes the distance. The probe will then move clear of the obstruction by a pre-set distance.

Details of suitable methods of installing test set-up programmes for different test procedures and methods of calibration of the load cell (required for force measurement) and the probe height are provided by the instrument. The instrument should be calibrated after installation, whenever it is moved, and afterwards at monthly intervals. The probe height should be calibrated whenever the instrument is set to carry out the procedures of the Bloom test (see clause 7), whenever the probe is changed, and when a “stop” condition occurs during a test.

The instrument can read out a force value (which may be calibrated in grams Bloom) on the console keyboard display, or may be connected to a computer.

This arrangement makes it possible to show data in graphical format, observe multiple peaks, measure gradients, areas and averages, and save the data on disk. Several saved graphs can be recalled on screen and viewed together, and average values shown and saved. From this information, the user can establish the repeatability and statistical accuracy of the data obtained.



Key

1	Probe assembly	13	Main circuit P/C board plug
2	Bidirection load cell	14	Display panel P/C board plug
3	Lead screw	15	Transformer
4	Support rods	16	Upper limit switch
5	Load cell mounting block	17	Lower limit switch
6	Load cell mounting block guide rod	18	Nut under cap cover
7	Stepping motor	19	Securing nuts for holding display board
8	Probe assembly swivel joint	20	Internal adjustment trimmers
9	Tube for protection of probe rod	20-1	Overload level
10	Display panel P/C board	20-2	Trigger level
11	Main circuit P/C board	20-3	Front panel zero control offset
12	3 mm grub screw	20-4	Cell gain

Figure A.1 — Stevens LFRA texture analyser gelometer — Front control panel and internal side views

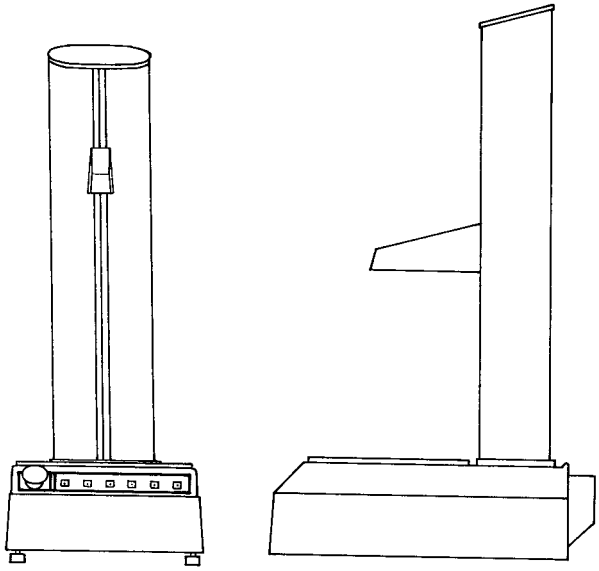


Figure A.2 — Stevens QTS 25 materials evaluation apparatus — Front and side views

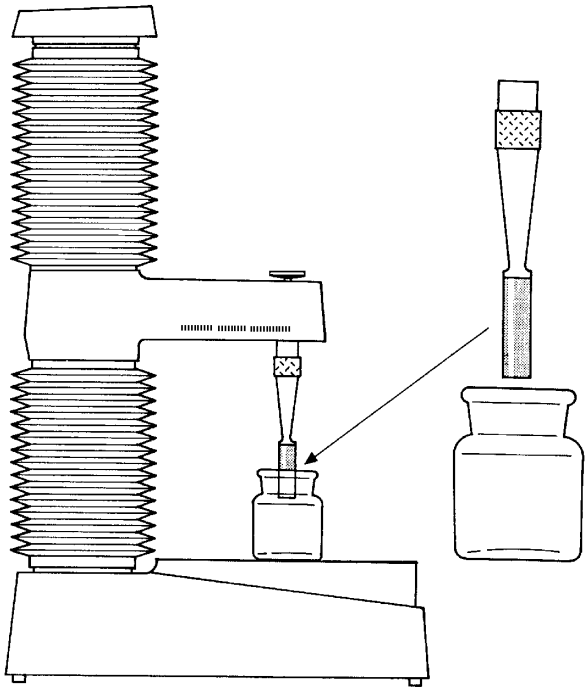


Figure A.3 — Stable Micro Systems TA-XT2 texture analyser — Side view showing probe position and jelly-testing jar

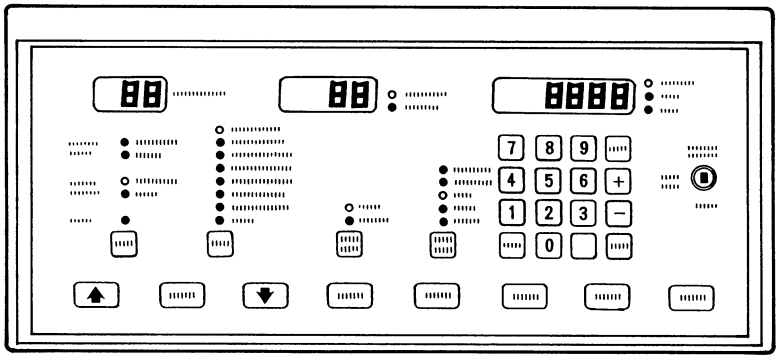


Figure A.4 — Stable Micro Systems TA-XT2 texture analyser — Diagrammatic view of control console

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