BRITISH STANDARD

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12092:2001

Adhesives — Determination of viscosity

The European Standard EN 12092:2001 has the status of a British Standard

ICS 83.180

National foreword

This British Standard is the official English language version of EN 12092:2001.

The UK participation in its preparation was entrusted to Technical Committee PRI/52, Adhesives, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

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EN 12092

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English version

Adhesives - Determination of viscosity

Adhésifs - Détermination de la viscosité

Klebstoffe - Bestimmung der Viskosität

This European Standard was approved by CEN on 30 September 2001.

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Contents

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 193 "Adhesives", the secretariat of which is held by AENOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2002, and conflicting national standards shall be withdrawn at the latest by April 2002.

In this standard the Annex A is informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies six methods which, experience has shown, are suitable for measuring the viscosity of a wide range of adhesives:

- rotational viscometer method;

- cone and plate viscometer method;

- falling ball viscometer method;

- glass capillary viscometer method;

- flow cup method;

- pressurised extrusion method.

This standard does not intend to describe equipment, which is unique to one manufacturer, rather it describes generic types of viscometer. The instruments described operate on very different principles making it inadvisable to compare results from one type of viscometer with those from another. However, results from different instruments of the same type should be comparable.

NOTE 1 Some modern viscometers incorporate integrated electronic measuring devices and have a high degree of automation. The user will not necessarily have access to (or need to know) full details of an instrument's measuring systems. However, all viscometers are capable of being calibrated and, to conform to this standard, the viscometer used should be calibrated in the appropriate viscosity range before a test. Certified calibration fluids should be used for this purpose and evidence of calibration given with the test results.

NOTE 2 The properties of many adhesives are very sensitive to test conditions. It is important therefore that the test report contains comprehensive information on the conduct of the test.

2 Normative references

This European Standard incorporates by dated or undated reference provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 923	*Adhesives — Terms and definitions.*
EN 1066	*Adhesives – Sampling.*
EN 1067	*Adhesives — Determination and preparation of samples for testing.*
EN ISO 2431	*Paints and varnishes — Determination of flow time by use of flow cups (ISO 2431:1993, including Technical corrgendum 1:1994).*
EN ISO 2555	*Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield test method (ISO 2555:1989).*
EN ISO 3104	*Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity (ISO 3104:1994).*

EN ISO 3219	*Plastics — Polymers/resins in the liquid or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate (ISO 3219:1993).*
ISO 3105	*Glass capillary kinematic viscometers — Specifications and operating instructions.*
ISO 4575	*Plastics — Polyvinyl chloride pastes — Determination of apparent viscosity using the Severs viscometer.*
ISO/DIS 12058-2	*Plastics — Determination of viscosity using a falling ball viscometer – Part 2: Free-falling-ball method.*

3 Terms and definitions

For the purposes of this European Standard the following term and definition, together with those given in EN 923, apply.

3.1
viscosity
property of a material to resist deformation increasingly with increased rate of deformation

4 Choice of the preferred method

The preferred method shall be chosen according to the types and requirements of the adhesives used.

For Newtonian liquids with viscosity below 10 Pa·s, the method described in 6.5 is preferred. The alternative methods are given in 6.4 and 6.6.

For Newtonian liquids with a viscosity higher than 10 Pa·s, the methods described in 6.2 and 6.7 are preferred.

For non-Newtonian liquids the method described in 6.3 is preferred. The method given in 6.2 can also be used.

NOTE Newtonian liquid or ideal liquid is defined as a liquid characterized by a constant value for the shear stress divided by the rate of shear in simple shear flow and with zero normal stress differences.

Non-Newtonian liquid or non-ideal liquid is defined as a liquid not exhibiting Newtonian behaviour.

5 Safety

Persons using this standard shall be familiar with normal laboratory practice.

This standard does not purport to address all the safety problems, if any, associated with its use.

It is the responsibility of the user to establish health and safety practices and to ensure compliance with any European and national regulatory conditions.

6 Test methods

6.1 General

All the procedures described in the following test methods shall be carried out in duplicate. The difference between the values of two consecutive tests shall be less than 5% and less than 3% for the test method described in 6.2, in particular. If this is not the case, further tests shall be carried out until this requirement is fulfilled.

6.2 Rotational viscometer method

6.2.1 Principle

A spindle, usually in the shape of a cylinder or disc, rotates at a constant speed in the adhesive. The instrument measures the torsional resistance of rotation. Factors, which depend on the speed of rotation and the characteristics of the spindle, are applied to the torsional resistance measurement resulting in an estimate of the coefficient of viscosity.

The method is often applied to non-Newtonian fluids and the viscosity measured depends on the rate of shear.

A detailed description of the apparatus to be used and further details of this method are given in EN ISO 2555 and EN ISO 3219.

6.2.2 Procedure

6.2.2.1 Prepare the adhesive in accordance with the manuacturer's instructions. Unless otherwise specified, take a sample of the adhesive in accordance with EN 1066 and examine and prepare it in accordance with EN 1067.

6.2.2.2 Set up, calibrate and operate the viscometer in accordance with the manufacturer's instructions. Spindles and speeds shall be chosen such that the instrument reading is in the range of 20% to 95% of the full scale value.

6.2.2.3 Ensure that the temperature of the test specimen is within the prescribed limits. Unless otherwise specified, the temperature of the test specimen shall be maintained at (23±0,5)°C. Heat or cool the container holding the test specimen by immersion in a thermostatically controlled bath. Special attachments are usually available for testing hot melt adhesives.

6.2.2.4 Start the motor and leave the spindle turning in the adhesive until a steady value is recorded. In some fluids the reading of the instrument will not settle, but will continue to drift. In such cases, record the reading after a specific time.

6.2.2.5 Stop the motor, wait until the spindle has stopped, then restart the motor and take a second measurement.

6.2.2.6 Repeat the process described in 6.2.2.5 until two consecutive readings differ by no more than 3%. Take the average of these two values.

6.2.2.7 After each determination, remove the spindle from the appliance and wash it carefully in the appropriate solvent.

6.2.3 Expression of results

Calculate the estimate of viscosity by the method prescribed by the manufacturer of the viscometer.

Express the result to three significant figures in units of Pa·s or in mPa·s as appropriate.

If required, plot the characteristic viscosity/shear rate curve for the adhesive.

6.2.4 Test report

The test report shall contain, at least, the following information:

a) reference to this European Standard;

b) identification of the product tested including details of any mixing or other preparations carried out;

c) the test temperature;

d) details of the viscometer, manufacturer and model, including the spindles and speeds used and the shear rate, if known;

e) in cases where a measurement was taken after a specific time, the value of this time period;

f) the value(s) of the viscosity expressed as indicated in 6.2.3;

g) proof of instrument calibration;

h) the test date.

6.3 Cone and plate viscometer method

6.3.1 Principle

The adhesive under test is contained between the conical end of a rotating disc and a static plate. The viscosity of the adhesive is related to the torque required to maintain a given speed of rotation and the diameter and angle of the cone.

A detailed description of the apparatus to be used and further details on this method are given in EN ISO 3219.

6.3.2 Procedure

6.3.2.1 Prepare the adhesive in accordance with the manufacturer's instructions. Unless otherwise specified, take a sample of the adhesive in accordance with EN 1066 and examine and prepare it in accordance with EN 1067.

6.3.2.2 Set up and calibrate the viscometer in accordance with the manufacturer's instructions.

Unless otherwise stated the instrument shall be adjusted to a shear rate of 18 000 s^{-1}.

6.3.2.3 With the cone in the operating position but with no adhesive in place, ensure that the viscometer reaches the prescribed temperature. Unless otherwise specified, the test shall be carried out at (23 $\pm$ 0,5)°C.

6.3.2.4 Start the cone rotating and zero the instrument.

6.3.2.5 Stop the rotation and raise the cone to a high position.

6.3.2.6 Place a small amount of the adhesive (as specified by the viscometer manufacturer) in the centre of the plate and lower the cone again to the operating position.

6.3.2.7 Remove excess adhesive.

6.3.2.8 Wait approximately 15 s for the sample to reach the test temperature.

6.3.2.9 Start the cone rotating. Allow the reading to reach a steady value and record the reading.

6.3.2.10 After each determination carefully clean all components with an appropriate solvent.

6.3.3 Expression of results

Calculate the estimate of viscosity by the method prescribed by the manufacturer of the viscometer.

Express the result to three significant figures in units of Pa·s or in mPa·s as appropriate.

If required, plot the characteristics viscosity/shear rate curve for the adhesive.

6.3.4 Test report

The test report shall contain, at least, the following information:

a) reference to this European Standard;

b) identification of the product tested including details of any mixing or other preparations carried out;

c) the test temperature;

d) details of the viscometer, manufacturer and model, including the spindles and speeds used and the shear rate, if known;

e) in cases where a measurement was taken after a specified time, the value of this time period;

f) the value(s) of the viscosity expressed as indicated in 6.3.3;

g) proof of instrument calibration;

h) the test date.

6.4 Falling ball viscometer method

6.4.1 Principle

The time for a ball of known density to fall through a known distance in a column of adhesive is used to estimate the viscosity of the adhesive. The tube containing the adhesive is contained within a thermostatically controlled sleeve. The measurement range of the instrument can be extended by using a series of standardized balls and tubes.

A detailed description of the apparatus to be used and further details on this method are given in ISO/DIS 12058-2.

6.4.2 Procedure

6.4.2.1 Prepare the adhesive in accordance with the manufacture'r instructions.

Unless otherwise specified, take a sample of the adhesive in accordance with EN 1066 and examine and prepare it in accordance with EN 1067.

6.4.2.2 Set up and calibrate the viscometer in accordance with the manufacturer's intructions.

6.4.2.3 Ensure that the temperature of the test chamber is within prescribed limits. Unless otherwise specified, the test specimen shall be maintained at (23 ± 0,5)°C.

6.4.2.4 Introduce the test specimen into the calibrate tube together with the chosen ball.

6.4.2.5 Close off the tube with the stopper and condition for 30 min. This will allow the temperature of the specimen to approach that of the thermostatically controlled sleeve.

6.4.2.6 Invert the tube and allow the ball to drop. Measure the time to the nearest 0,1 s for the ball to fall between the two timing marks.

6.4.2.7 After each determination, remove the tube from the appliance and clean it carefully with an appropriate solvent.

6.4.3 Expression of results

Calculate the estimate of viscosity by the method prescribed by the manufacturer of the viscometer.

Express the result to three significant figures in units of Pa·s or in mPa·s as appropriate.

6.4.4 Test report

The test report shall contain, at least, the following information:

a) reference to this European Standard;

b) identification of the product tested including details of any mixing or other preparations carried out;

c) the test temperature;

d) details of the viscometer, including the tubes and balls used;

e) the value(s) of the viscosity expressed as indicated in 6.4.3;

f) proof of instrument calibration;

g) the test date.

6.5 Glass capillary viscometer method for the determination of kinematic viscosity

6.5.1 Principle

The time is measured for a fixed volume of a liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature.

A detailed description of the apparatus to be used and further details on this method are given in EN ISO 3104 and ISO 3105.

6.5.2 Procedure

6.5.2.1 Prepare the adhesive in accordance with the manufacturer's instructions.

Unless otherwise specified, take a sample of the adhesive in accordance with EN 1066 and examine and prepare it in accordance with EN 1067.

6.5.2.2 Proceed in accordance with EN ISO 3104 and ISO 3105. If necessary, run a set of calibration fluids before proceeding with the testing of the adhesives.

6.5.3 Expression of results

Calculate the estimate of viscosity by the method prescribed by the manufacturer of the viscometer.

Express the result to three significant figures of Pa·s or in mPa·s as appropriate.

6.5.4 Test report

The test report shall contain, at least, the following information:

a) reference to this European Standard;

b) identification of the product tested including details of any mixing or other preparations carried out;

c) the test temperature;

d) details of the viscometer used;

e) the value(s) of the viscosity expressed as indicated in 6.5.3;

f) proof of instrument calibration;

g) the test date.

6.6 Flow cup method

6.6.1 Principle

The adhesive to be tested fills a standardized cup which has an accurately defined hole in its base. The hole is initially blocked to prevent the adhesive escaping. The time which elapses between the product being released through the hole and the moment at which the flow ceases to become continuous can be converted to an estimate of viscosity.

Reproducible results are only possible with Newtonian or near-Newtonian fluids.

A very wide range of flow cups are available and are usually calibrated for time against kinematic viscosity. Some experimentation can be necessary in order to select the best cup for a particular determination. The aim is to choose a flow cup that will result in a flow time of 20 s to 100 s.

A detailed description of the apparatus to be used and further details on this method are given EN ISO 2431.

6.6.2 Procedure

6.6.2.1 Prepare the adhesive in accordance with the manufacturer's instructions. Unless otherwise specified, take a sample of the adhesive in accordance with EN 1066 and examine and prepare it in accordance with EN 1067. Allow the sample to stand until all bubbles have dispersed.

6.6.2.2 Set up a clean, dry cup on its stand in a draught-free position. Level the cup and ensure that the temperature is within the specified range. Unless otherwise specified, the temperature throughout the test shall be maintained at (23 ± 0,5)°C.

6.6.2.3 Place a suitable empty container beneath the flow cup so that the distance between the orifice of the cup and the base of the container does not exceed 100 mm.

6.6.2.4 With the finger of the operator blocking the orifice of the cup, fill the cup with the sample until excess adhesive just overflows into the cup gallery. Allow the bubbles to disperse and eliminate any meniscus with a levelling straight edge.

6.6.2.5 Withdraw the finger from the orifice and simultaneously start the stop-watch. Stop the watch as soon as the flow in the vicinity of the orifice ceases to be continuous. Record the flow time to nearest 0,5 s.

6.6.2.6 Carry out a second determination using another part of the originally prepared sample. If the first and second values differ by more than 5% from their mean value then carry out a third determination. If the third determination and one of the two previous determinations do not differ by more than 5% from their mean value then eliminate the outlying value and take the mean of the two remaining values as the result of the test.

6.6.2.7 If is not possible to obtain two results which differ by less than 5% then the method shall be considered as unsuitable for the adhesive under test and an alternative method shall be considered.

6.6.3 Expression of results

Express the result in seconds.

6.6.4 Test report

The test report shall contain, at least, the following information:

a) reference to this European Standard;

b) identification of the product tested details of any mixing or other preparations carried out;

c) the test temperature;

d) details of the cup used;

e) the value(s) of the viscosity expressed as indicated in 6.6.3;

f) proof of instrument calibration;

g) the test date.

6.7 Pressurised extrusion method for the determination of viscosity of paste adhesives

6.7.1 Principle

Measurement of the time to extrude (at a known pressure) a known mass of paste adhesive through a standard die enables an estimate to be made of the shear rate and dynamic viscosity.

A certain amount of experimentation may be necessary to select the appropriate die and extrusion pressure. The aim shall be to extrude a sample of approximately 10 g in 1 min to 2 min.

The method can be used to determine the viscosity at one particular shear rate or to prepare a full viscosity/shear rate profile of the adhesive.

A detailed description of the apparatus to be used and further details on this method are given in ISO 4575.

6.7.2 Procedure

6.7.2.1 Prepare the adhesive in accordance with the manufacturer's instructions.

Unless otherwise specified, take a sample of the adhesive in accordance with EN 1066 and examine and prepare it in accordance with EN 1067.

6.7.2.2 Set up and calibrate the viscometer in accordance with the manufacturer's instructions.

6.7.2.3 Ensure that the temperature of the test chamber is within prescribed limits. Unless otherwise specified, the test specimen shall be maintained at $(23 \pm 0{,}5)$°C.

6.7.2.4 Set the test pressure, operate the viscometer and extrude a sample into a beaker on a balance. Use a stop-watch to measure the time (to the nearest 0,1 s) to extrude approximately 10 g. Express the actual weight extruded to the nearest 0,1 g.

6.7.2.5 Repeat the procedure described in 6.7.2.4 to obtain two further determinations at the same pressure setting.

6.7.2.6 If a full study of viscosity versus shear rate is required, reset the test pressure and repeat the procedure given in 6.7.2.4 and 6.7.2.5 as required.

6.7.3 Expression of results

Calculate the estimate of viscosity by the method prescribed by the manufacturer of the viscometer.

Express shear rate in s^{-1} and viscosity in Pa·s

If required, plot the characteristics viscosity/shear rate curve for the adhesive.

6.7.4 Test report

The test report shall contain, at least, the following information:

a) reference to this European Standard;

b) identification of the product tested including details of any mixing or other preparations carried out;

c) the test temperature;

d) details of the viscometer used, including dies and test pressures;

e) the value(s) of the viscosity expressed as indicated in 6.7.3;

f) proof of instrument calibration;

g) the test date.

Annex A
(informative)

Dynamic and kinematic viscosity. Definition and calculation

Two measures of viscosity are described: dynamic and kinematic viscosity. It is necessary to know the density of the adhesive to convert from one measure to the other.

Dynamic viscosity (η_d) is defined as shear stress divided by the rate of shear in steady flow. It can be derived from Newton's law of viscous flow which states that in a perfectly fluid material the shear rate is proportional to the shearing force applied, as shown in equation 1:

$$F = \eta_d \, A \frac{dv}{dx} \qquad (1)$$

where

F is the tangential force between two parallel planes of area A, spaced dx apart, moving with a relative velocity of dv.

From equation (1); $\eta_d = \frac{F}{A}\frac{dx}{dv}$

η_d has the SI dimensions of $\frac{N}{m^2} \cdot \frac{m}{m/s} = Pa \cdot s$

It is sometimes more convenient to use millipascal seconds (mPa.s).

Kinematic viscosity is defined as dynamic viscosity divided by the density of the material measured at the same temperature. It has the SI dimension of $m^2 \cdot s^{-1}$ but, for convenience, it is sometimes expressed in $mm^2 \cdot s^{-1}$.

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