INTERNATIONAL STANDARD

ISO 12058-1

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Plastics — Determination of viscosity using a falling-ball viscometer —

Part 1:
Inclined-tube method

Plastiques — Détermination de la viscosité au moyen d'un viscosimètre à chute de bille —

Partie 1: Méthode du tube incliné

Reference number
ISO 12058-1:1997(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 12058-1 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties.*

It cancels and replaces annex B of ISO 9371:1990.

ISO 12058 consists of the following parts, under the general title *Plastics — Determination of viscosity using a falling-ball viscometer*:

— *Part 1: Inclined-tube method*

— *Part 2: Free-falling-ball method*

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Plastics — Determination of viscosity using a falling-ball viscometer —

Part 1:
Inclined-tube method

1 Scope

This part of ISO 12058 specifies the general principles of a method, using an inclined-tube falling-ball viscometer, for determining the viscosity of polymers and resins in the liquid emulsified or dispersed state. It is intended for application to liquids over a viscosity measurement range of 0,6 mPa·s to 250 000 mPa·s (temperature range – 20 °C to + 120 °C) for which the shear stress and shear rate are proportional, i.e. the viscosity is independent of the shear rate. This ideal behaviour is commonly known as Newtonian behaviour. If a liquid differs significantly from this behaviour, different results may be obtained with the different balls of a falling-ball viscometer or from viscometers with different geometries, such as capillary and rotational viscometers.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 12058. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this part of ISO 12058 are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2811-1:—[1], *Paints and varnishes — Determination of density — Part 1: Pyknometer method.*

3 Principle

The viscosity of a liquid is determined by observing the motion of a solid sphere under the influence of gravity in an inclined cylindrical tube filled with the liquid.

4 Measurand and units

Dynamic viscosity, expressed in millipascal seconds (mPa·s).

1) To be published. (Revision of ISO 2811:1974)

5 Measurement range and temperature range

Viscosity-measurement range:	0,6 mPa·s to 250 000 mPa·s
Minimum falling time for ball:	60 s for ball No. 1 [2] 50 s for balls 2 to 4 30 s for balls 5 and 6
Temperature range:	– 20 °C to + 120 °C

6 Apparatus

6.1 Falling-ball viscometer[3] (see figure 1)

The apparatus consists of an inclined measurement tube (falling-ball tube) filled with the liquid under test and made of thermally aged, calibrated, precision borosilicate-glass tubing with a coefficient of linear expansion of $3{,}3 \times 10^{-6}$ °C^{-1}, plus six balls of diameter 15,81 mm to 11,0 mm (see table 1), each of balls 1 to 4 having the same coefficient of expansion as the tube itself. To minimize errors, the measurement tube and balls shall be free of flaws.

Table 1 — Balls for use in falling-ball viscometer having a measurement tube with an inside diameter of 15,94 mm ± 0,01 mm

Ball No.	Material	Density, ρ_1 (typical) g/cm^3	Diameter mm	Out-of-roundness mm	Constant K (typical) mPa·s·cm^3/(g·s)	Dynamic-viscosity measurement range (typical) mPa·s
1	Borosilicate glass	2,4	15,81 ± 0,01	± 0,000 5	0,007	0,6 to 10
2	Borosilicate glass	2,4	15,60 ± 0,05	± 0,000 5	0,09	7 to 130
3	Nickel-iron	8,1	15,60 ± 0,05	± 0,001	0,09	30 to 700
4	Nickel-iron	8,1	15,2 ± 0,1	± 0,001	0,7	200 to 4 800
5	Nickel-iron or steel	7,7 to 8,1	14,0 ± 0,5	± 0,001	7	1 500 to 45 000
6	Steel	7,7 to 7,8	11 ± 1	± 0,002	35	> 7 500

For a given ball, the calibration constant K of the apparatus depends on the internal diameter of the tube. The values given in table 1 apply to a tube 15,94 mm ± 0,01 mm in diameter.

The measurement tube has two circular marks defining the measurement distance to within 100 mm ± 1 mm. The tube is surrounded by a tubular glass jacket for temperature control and fixed to a stand in such a manner that the axis of the tube is inclined at 10° ± 1° to the vertical for measurement purposes[4]. The measurement tube and the glass jacket can be inverted by rotating them together about their mounting point on the stand in order to return the ball to the starting position. The ends of the measurement tube are closed with two plugs, one of which contains a capillary joined to a hollow space. This type of closure prevents unacceptable pressure variations occurring, as well as the entry of air due to temperature fluctuations. The liquid under test is completely surrounded by the jacket and plugs, thus preventing evaporation or the formation of a surface skin. The stand is equipped with a spirit level and feet with levelling screws. A removable thermometer is fitted (see 6.2).

Each viscometer shall have a test certificate, issued by the manufacturer or by a third party, stating the individual apparatus constants. The viscometer shall be recalibrated whenever a new measurement tube or a new ball is used or when considerable demands are placed on the viscometer (e.g. a series of measurements on aggressive liquids).

2) See table 1.

3) The determination of viscosity using an inclined-tube falling-ball viscometer may be carried out using equipment supplied by a number of manufacturers. One example of such an instrument is the Hoeppler viscometer as described in DIN 53015:1978, *Viscometry; Measurement of viscosity using the Hoeppler falling-ball viscometer.*

4) Apparatus with an inclination other than 10° is not suitable for use with this part of ISO 12058.

Dimensions in millimetres

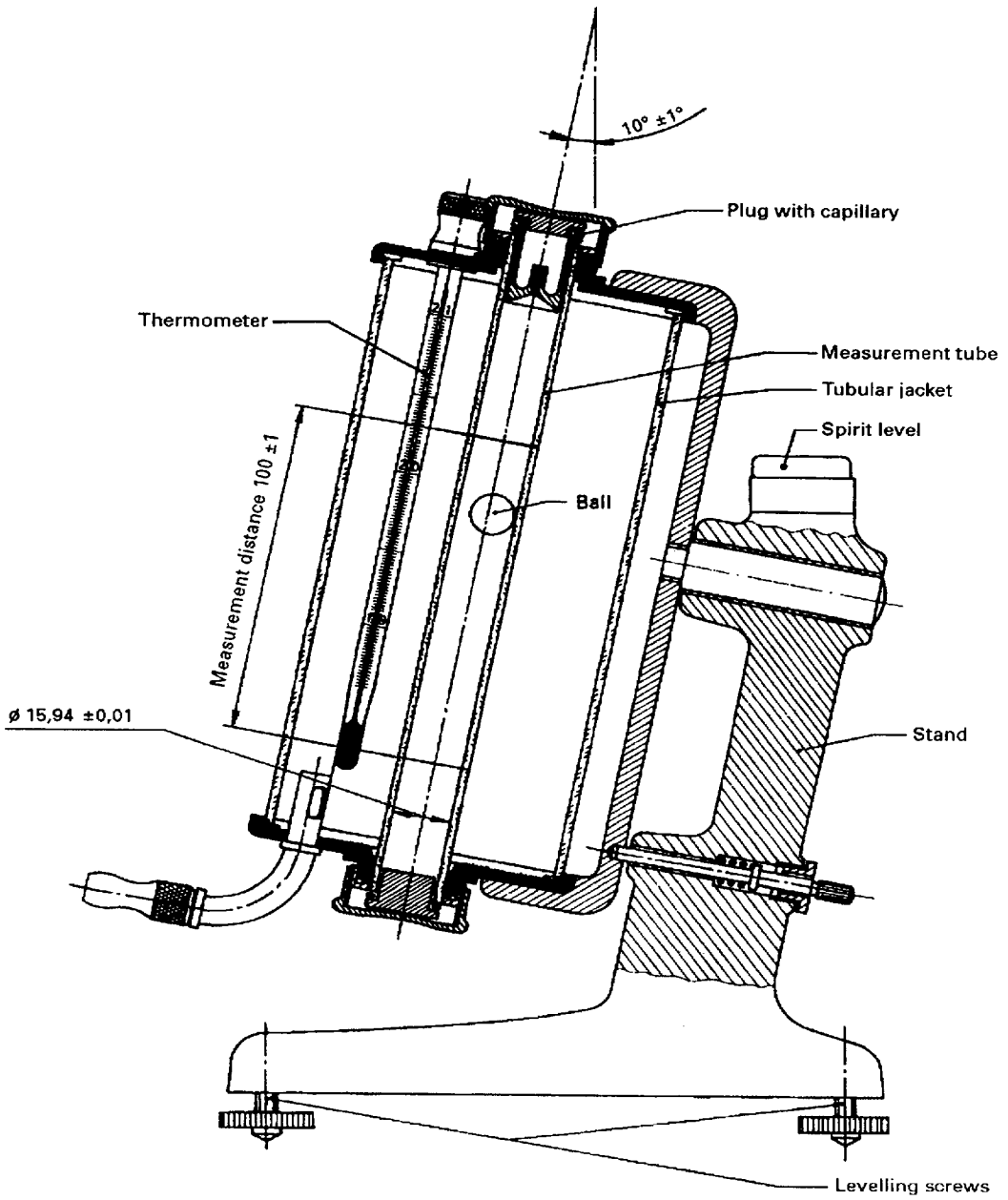

Figure 1 — Typical falling-ball viscometer

6.2 Thermometer

The special thermometer used in the apparatus is placed in the liquid in the thermal jacket. It shall have a scale graduated at intervals of not more than 0,1 °C. It shall be calibrated totally immersed, and shall be capable of measuring the temperature to ± 0,03 °C. The thermometer reading shall be corrected as indicated in the calibration certificate. The thermometer shall be protected against radiant heat during the measurement.

6.3 Timer

The time taken by the ball to travel the distance between the two circular marks shall be measured using one of the following.

6.3.1 Stopwatch, having a scale interval not greater than 0,1 s. The stop watch shall be compared from time to time with a standard clock accurate to within 15 s in 24 h. It is advisable to use calibrated stopwatches.

6.3.2 Electronic timer, based on frequency counting or comprising synchronous clocks. The frequency used shall be constant to within 10^{-4} of its normal value.

6.3.3 Automatic timer, with the same accuracy as the stopwatch (6.3.1).

6.4 Thermostat

The temperature in the jacket shall be maintained constant, by means of a thermostat, to within at least ± 0,03 °C over the temperature range from 10 °C to 80 °C and to within ± 0,05 °C at temperatures outside this range. Only fluorescent tubes, which radiate little heat, shall be used to illuminate the apparatus.

7 Sampling

Sampling shall be carried out in accordance with the specification or standard for the liquid under test.

8 Procedure

Ensure that all parts of the apparatus coming into contact with the liquid under test are clean[5] and dry.

Pour the liquid under test into the measurement tube and introduce the ball. Then place the plug with the capillary in the upper end of the measurement tube and screw tight. The ball shall not have any bubbles adhering to it. Maintain the liquid under test at the specified test temperature for at least 15 min[6]. Before each series of measurements, roll the ball along the length of the tube once in order to ensure thorough mixing of the liquid. Then measure the time the ball takes to travel between the two circular marks (see figure 1).

Repeat the measurement at least three times.

If the density of the liquid under test is not known, determine it in accordance with ISO 2811-1.

9 Expression of results

Calculate the dynamic viscosity η, expressed in millipascal seconds, using the following equation:

$$\eta = K(\rho_1 - \rho_2)\, t$$

where

K is the calibration constant of the instrument (see table 1 and 6.1);

ρ_1 is the density, in grams per cubic centimetre, of the ball (see table 1);

ρ_2 is the density, in grams per cubic centimetre, of the liquid under test;

t is the time, in seconds, for the ball to travel the distance between the two marks.

Take as the result the average, rounded to three significant figures, of the determinations carried out.

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5) Usually the tube is cleaned by rinsing it with a suitable solvent.

6) If highly viscous liquids are measured or if the measurement temperature differs by more than about 20 °C from room temperature, equilibration may require significantly longer than 15 min.

10 Precision

To assess the reliability of results, the following principles are used:

Repeatability (same operator, same apparatus, same procedure)

If two results are obtained under repeatability conditions, both results may be regarded as acceptable and in accordance with this part of ISO 12058 if they differ from their mean value by not more than the appropriate limit given in table 2.

Reproducibility (different operators, different apparatus, but same procedure)

If a result is obtained under reproducibility conditions in each of two different laboratories, both results may be regarded as acceptable and in accordance with this part of ISO 12058 if they do not differ from their mean value by more than the appropriate limit given in table 2.

Table 2 — Repeatability and reproducibility limits

Ball No.	Repeatability limit, %	Reproducibility limit, %
1	1,0	2
2 to 4	0,5	1
5	0,7	1,5
6	1,5	3

The values given in this table were established following tests performed in Germany using DIN 53015:1978, *Viscometry; Measurement of viscosity using the Hoeppler falling-ball viscometer.*

11 Test report

The test report shall include the following information:

a) the reference number of this part of ISO 12058;
b) all details necessary for complete identification the material under test;
c) the date of sampling;
d) the test temperature, in degrees Celsius;
e) the average dynamic viscosity, in millipascal seconds;
f) any deviations from the procedure specified;
g) any unusual features noted during the determination;
h) the date of the test;
i) the name and signature of the person responsible for the determination.

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