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Bitumen and bituminous binders — Determination of efflux time by the efflux viscometer

Part 1: Bituminous emulsions

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Bitumen and bituminous binders - Determination of efflux time by the efflux viscometer - Part 1: Bituminous emulsions

Bitumes et liants bitumineux - Détermination du temps d'écoulement à l'aide d'un viscosimètre à écoulement - Partie 1: Emulsions de bitume

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Ausflusszeit mittels Ausflussviskosimeter - Teil 1: Bitumenemulsionen

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Foreword

This document (EN 12846-1:2011) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by September 2011, and conflicting national standards shall be withdrawn at the latest by September 2011.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 12846:2002.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive 89/106/EEC.

This European Standard EN 12846 consists of the following parts under the general title *Bitumen and bituminous binders – Determination of efflux time by the efflux viscometer:*

— *Part 1: Bituminous emulsions;*

— *Part 2: Cut-back and fluxed bituminous binders.*

EN 12846-1 has been created as the result of the merging of EN 12846:2002 and EN 13357:2002 under a single EN 12846 reference (two different parts), since both standards describe very similar procedures with identical equipment. The two different parts have been made as consistent as possible by eliminating all existing minor differences between both methods.

Compared with the previous edition, EN 12846:2002, the scope and principle were detailed, Figure 2 was added, tolerances were specified or modified in the apparatus dimensions, details were added in the test procedure and measurements made and the test precision data were reviewed.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a method for the determination of the efflux time at 40 °C of bituminous emulsions in seconds using an efflux viscometer. Alternative test temperature is 50 °C.

NOTE The procedure described in this standard may also be followed to determine efflux time at other temperatures such as for instance 25 °C.

WARNING — The use of this European Standard may involve hazardous materials, operations and equipment. This European Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this European Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 58, *Bitumen and bituminous binders — Sampling bituminous binders*

EN 12594, *Bitumen and bituminous binders — Preparation of test samples*

EN 13302, *Bitumen and bituminous binders — Determination of dynamic viscosity of bituminous binder using a rotating spindle apparatus*

EN ISO 4788, *Laboratory glassware — Graduated measuring cylinders (ISO 4788:2005)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
viscosity
internal resistance of a fluid to flow

3.2
efflux time
time needed for a specified volume of a material to flow through a specified orifice at a specified temperature

NOTE The efflux time is an indirect measure of the viscosity and is also referred to as “pseudo-viscosity”.

4 Principle

The efflux time of a bituminous emulsion is determined using an efflux viscometer known as the Standard Tar Viscometer (STV) which determines the time of efflux of a 50 ml sample through a 10 mm or a 4 mm or a 2 mm orifice at a specified temperature.

Whatever temperatures or orifice diameters used, the efflux time shall not exceed 600 s. For highly viscous emulsions, EN 13302 shall be used.

5 Reagents and materials

5.1 Light mineral oil.

Light mineral oil having a viscosity equal or lower than 7 mm^2/s at 40 °C.

5.2 Solution Sa.

Aqueous solution with a 1 % mass of sodium lauryl sulfate preferably, or aqueous solution with a nominal 1 % mass of sodium oleate shall be used for testing anionic emulsions. Those solutions (surfactants) shall be solubilised with 1 % mass of sodium hydroxide (NaOH). The actual soap solution may be used if available on site, in case of routine tests or for simplicity.

NOTE S_a stands for "anionic solution" as this solution is used for testing anionic emulsions.

5.3 Solution S_C.

Aqueous solution with a 1 % mass of alkyltrimethylammonium chloride preferably, or aqueous solution with a nominal 1 % mass of cetyltrimethylammonium bromide shall be used for testing cationic emulsions. Those surfactants shall be solubilised with 1 % mass of hydrochloric acid (HCl) (HCl concentration shall be approximately equal to 1 mol/l). The actual aqueous phase may be used if available on site, in case of routine tests or for simplicity.

NOTE 1 S_C stands for "cationic solution" as this solution is used for testing cationic emulsions.

NOTE 2 The solution of 1 % of cetyltrimethylammonium bromide should be prepared slightly above 25 ºC (but not higher than 30 ºC). Then it should be stored at a temperature of (25±1) ºC before test.

6 Apparatus

Usual laboratory apparatus and glassware, together with the following:

6.1 Efflux viscometer (see an example of viscometer on Figure 1) consisting essentially of a cup with an orifice in the centre of the base which may be closed by a ball-and-socket valve (see Figure 2).

Three forms are required, differing only in the size of the orifice (10 mm, 4 mm and 2 mm). For other dimensions of the cup and the ball-valve, see Figure 3. The cup cylinder shall be made of brass. The ball valve should be made of corrosion-resistant metal, with a ball on a rod, a levelling peg attached to the rod and a hemispherical top by means of which the valve may be supported in a vertical position.

The viscometer cups shall be equipped with suitable corks or caps for closing the orifices with the ball valve in position, and some means of covering the cups (e.g. lids) to prevent evaporation of water and minimize surface cooling effects.

The viscometer-cup holder shall be capable of:

— supporting one or more cups in a vertical position;

— providing a valve support to hold the valve at least 16 mm vertically above the orifice of the cup during efflux of the test material.

NOTE 1 To enhance resistance to wear and corrosion of the ball and socket valve, the bottom of the cup may be made from a different, corrosion resistant, material and screwed to the brass tube. It is then advised to use the same material, such as for instance phosphor-bronze, for the cup bottom and the ball valve. Wrought nickel alloy with copper or metals NiCu30 in accordance with ISO 9722 are possible materials for the rod of the ball valve.

NOTE 2 The viscometer cup should be provided with a lid suitable for closing the upper end of the cup without touching the test material when the cup is filled. This lid is provided with a central hole through which the thermometer can pass and with a groove on one side through which the rod of the valve can pass.

6.2 Viscometer water-bath, constant temperature for maintaining the test temperature to within ± 0,5 °C. A typical elevation and a plan of assembled viscometer are given in Figure 1.

6.3 Temperature controlled water bath, maintained at (40,0 ± 0,5) °C, in which one or more of the viscometer cups can be immersed up to the rim of the cup.

NOTE 1 Other test temperatures are (50,0 ± 0,5) °C or (25,0 ± 0,5) °C.

If a multiple-cup heating bath is used, the cups shall be separated from each other and from the walls of the bath by at least 55 mm.

A suitable support shall be provided to maintain the cup(s) in a vertical position.

An appropriate climatic chamber may be used for low breaking index emulsions (see 8.3.4).

NOTE 2 The viscometer water bath (6.2) may also be used to directly condition the test sample in the cup.

6.4 Thermometers, two, conforming to the requirements described in Annex A.

Other temperature measuring devices may be used instead of mercury stem thermometers. However, the mercury stem thermometer is the reference device. Therefore any alternative device employed shall be calibrated so as to provide the same readings as would be provided by the mercury stem thermometer, recognising and allowing for the fact of changed thermal response times compared with the mercury thermometer.

When measuring and controlling nominally constant temperatures, as in this test method, alternative devices can indicate greater cyclic variations than mercury thermometers, to an extent depending on the cycle time of heating and the power of the controlled heat input.

6.5 Receiver, consisting of a 100 ml cylinder with graduations at 20 ml, 25 ml and 75 ml, complying with the requirements of EN ISO 4788.

6.6 Timing device, capable of measuring the efflux time with an accuracy of ± 0,2 s.

7 Sampling

The material under test shall be sampled in accordance with EN 58 and prepared in accordance with EN 12594.

The test shall be carried out in duplicate.

8 Procedure

8.1 General

Carry out the procedure in laboratory at room temperature between 18 °C to 28 °C.

8.2 Preparation of apparatus

Clean the viscometer cup (6.1) with a suitable solvent, to remove any mark of binder, and thoroughly dry it to remove all traces of solvent. If necessary, rub the interior of the cup and/or clean the orifice. Use soft tissue-paper or some similar material that will not leave particles behind or abrade the metal.

When cleaning, care shall be taken not to damage the orifice.

8.3 Measurement

8.3.1 If the efflux time is unknown, measure it at 40 °C with the 4 mm orifice viscometer cup.

According to the efflux time obtained, 3 cases are possible. Choose the diameter of the orifice of the cup as follows.

1) If the efflux time is lower than 5 s, perform another determination at 40 °C with 2 mm orifice.

2) If the efflux time is greater than or equal to 5 s and lower than or equal to 600 s with still a continuous flow, report the value obtained at 40 °C with 4 mm orifice.

3) In case of a non continuous flow or if the efflux time is greater than 600 s, perform another determination at 50 °C with the 4 mm orifice or at 40 °C with the 10 mm orifice or by means of dynamic viscosity measurement (EN 13302).

Table 1 — Diameter of the orifice of the cup

Orifice size mm	**Efflux time** s	
	Minimum	Maximum
10, 4 or 2	5	Non continuous flow or 600 s

8.3.2 Condition the viscometer water-bath (6.2) and, if used, the water bath (6.3), by stirring the water in the bath with the relevant device and check that the temperature is at the required value for the test, maintained within ± 0,5 °C.

8.3.3 Close the lower part of the cup orifice with a cork or a cap and place the ball valve on top of the orifice. Carefully fill the cup with the prepared sample to such a height that the levelling peg on the valve is just immersed when the latter is vertical. Cover the top of the cup for example with a suitable lid. It shall be provided with a central hole and a groove on one side through which the rod of the valve (Figure 3, Key element 2) may be passed into the upper end of the cup. Pass the thermometer (6.4) through the central hole so that its bulb is approximately at the geometric centre of the sample.

8.3.4 Suspend the cup up to its rim in the water bath (6.3) or directly into the viscometer water bath (6.2) maintained within ± 0,5 °C of the test temperature for a period of time sufficient to reach the test temperature.

Due to emulsion instability, for low breaking index emulsions, preconditioning in the water bath (6.3) may be skipped. Preconditioning of the emulsion which has just been sampled and sieved (in accordance with EN 12594) in an appropriate receiver may be performed by directly placing this receiver in the climatic chamber (6.3). In that case, the viscometer-cup which is empty shall be heated in the same conditions.

8.3.5 If a separate water bath is used (6.3), remove the filled cup from the water bath and place it into the viscometer cup holder. Check that the sample is maintained at the required temperature. If not, wait till equilibrium at test temperature is reached again.

8.3.6 Remove any excess emulsion sample while removing the thermometer so that the final level of the binder is on the centre line of the levelling peg when the rod of the valve is in a vertical position. Remove the cork or stopper.

8.3.7 Pour the light mineral oil (5.1) or solution S_a (5.2) or solution S_c (5.3) into the receiver (6.5) up to the 20 ml graduation mark and place the receiver (6.5) directly under the orifice of the cup. Lift the valve and suspend it on the valve support such that the peg is levelled with the upper edge of the cup of at least 16 mm. Start the timing device (6.6) when the liquid in the receiver reaches the 25 ml graduation mark and stop it when the liquid reaches the 75 ml graduation mark.

Record the efflux time to the nearest 0,2 s.

8.3.8 Repeat Sampling (Clause 7) and Procedure (Clause 8) steps on a second emulsion test sample.

9 Expression of results

Express the result as the arithmetic mean of the two results obtained in accordance with Clause 8 to the nearest second, provided that individual results do not differ by more than the value for repeatability given in Table 2 under Clause 10.

If the two results differ by more than the above specified values, repeat the whole procedure.

10 Precision

10.1 General

The precision of the method was evaluated with 4 mm and 10 mm cups only. With the 2 mm cup, the precision is not available.

10.2 Repeatability

The difference between two successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values in only one case in twenty.

10.3 Reproducibility

The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values in only one case in twenty.

Table 2 — Precision

Efflux time s	**Repeatability**	**Reproducibility**
Below or equal to 20	1 s	2 s
above 20	5 % of the mean	10 % of the mean

11 Test report

The test report shall contain at least the following information:

a) type and complete identification of the sample under test (including date of the sampling and date of the sample preparation);

b) reference to this European Standard;

c) test temperature;

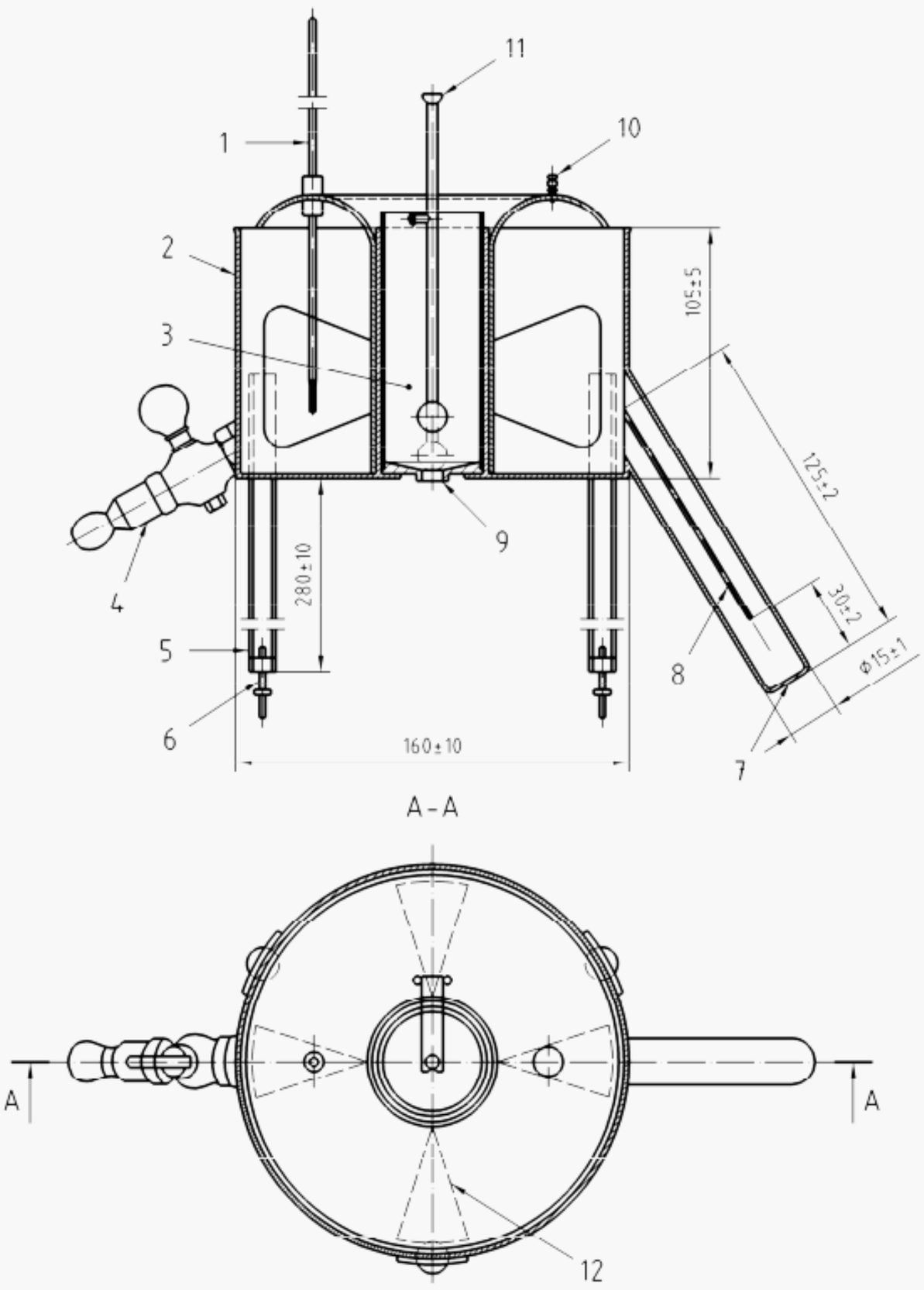
d) diameter of the orifice;

e) result of the test in seconds (see Clause 9);

f) any deviation, by agreement or otherwise, from the procedure specified;

g) date of the test.

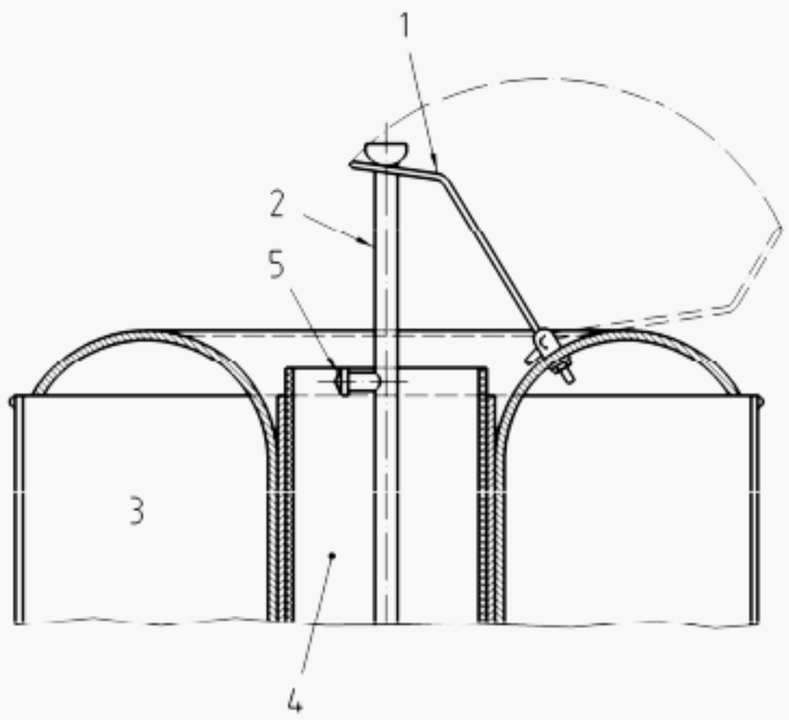
Dimensions in millimetres



Key

1. Thermometer
2. Water bath
3. Cup
4. Run off cock
5. Supporting legs
6. Levelling legs
7. Heating tube
8. Plate
9. Orifice
10. Insulted handle
11. Valve supported in "up" position
12. Vanes

Figure 1 — Typical elevation and plan of assembled viscometer (example)

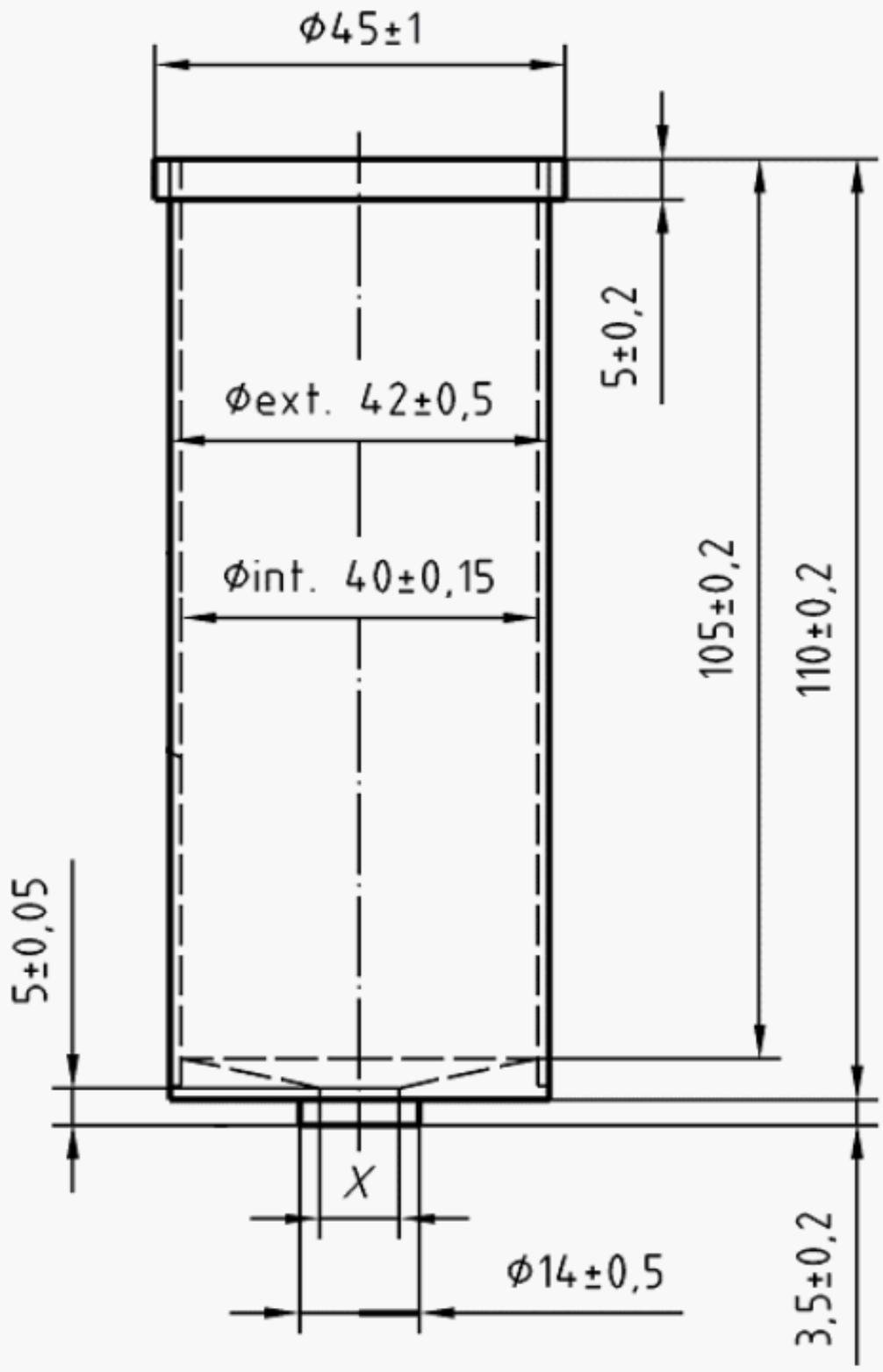


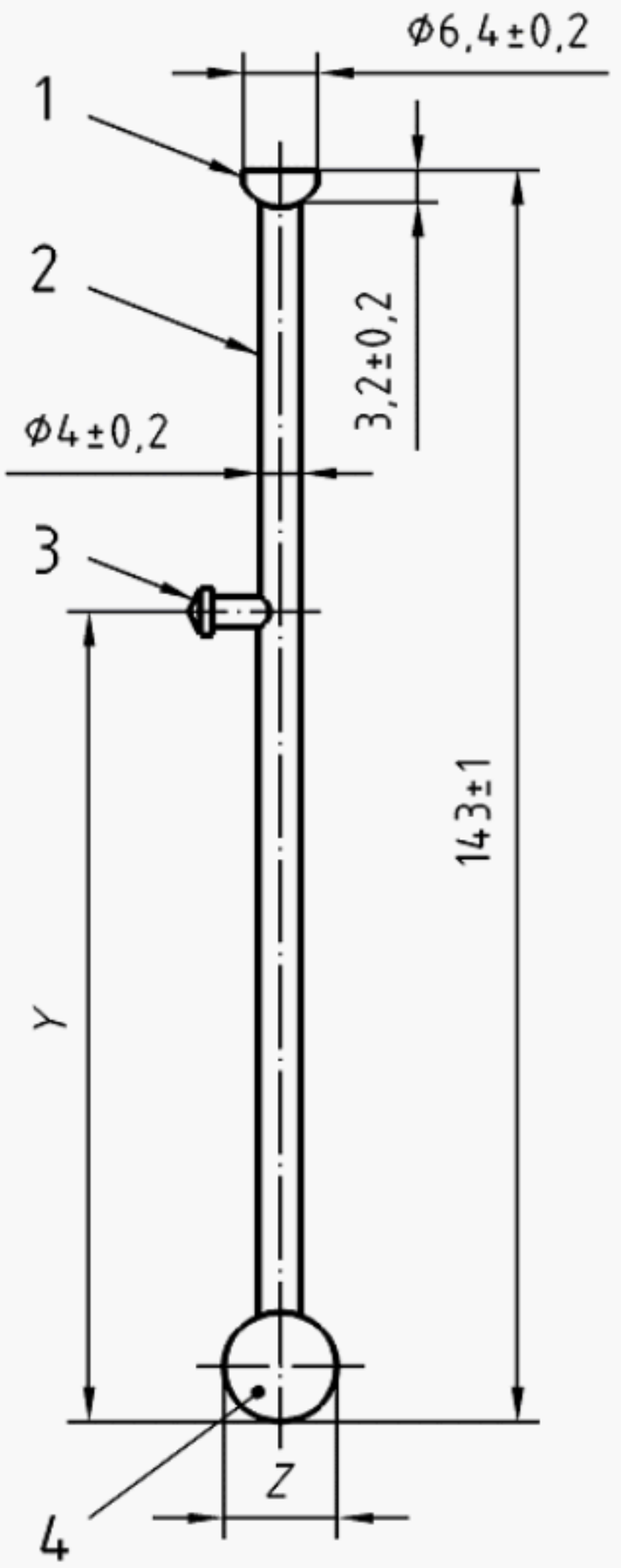
Key

1 Valve support
2 Rod of the valve
3 Water jacket
4 Viscometer cup
5 Levelling peg

Figure 2 — Typical section showing arrangement of the valve support (example)

Dimensions in millimetres





Key
1 Hemispherical top
2 Rod
3 Levelling peg
4 Ball

	X mm	*Y* mm	*Z* mm
2 mm cup	2,00 ± 0,025	90,0 ± 0,5	3,50 ± 0,05
4 mm cup	4,00 ± 0,05	90,3 ± 0,5	6,35 ± 0,05
10 mm cup	10,00 ± 0,05	92,0 ± 0,5	12,70 ± 0,05

Figure 3 — Viscometer-cup and ball valve

Annex A
(normative)

Specifications of thermometer

Temperature range	0 °C to + 45 °C or higher if needed
Immersion	65 mm
Scale marks:	
Subdivisions	0,2 °C
Long lines at each	1 °C and 5 °C
Numbers at each	5 °C
Maximum line width	0,15 mm
Scale error, max	0,2 °C
Expansion chamber permitting heating to	100 °C
Total length	330 mm to 350 mm
Stem outside diameter	5,5 mm to 8,0 mm
Bulb length	10 mm to 16 mm
Bulb outside diameter	not greater than stem
Scale location:	
Distance between bottom of bulb and line at 0 °C	100 mm minimum
Length of scale range	150 mm to 190 mm

NOTE 1 The thermometer IP 8C has been found suitable.

NOTE 2 Mercury thermometer ASTM 19C, respectively ASTM 17C, have been found suitable for carrying out viscosity measurements at 50 °C, respectively 25 ºC.

Bibliography

[1] ISO 9722, *Nickel and nickel alloys — Composition and forms of wrought products*

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