

English Version

## Bitumen and bituminous binders - Determination of the loss in mass after heating of industrial bitumen

Bitumes et liants bitumineux - Détermination de la perte de masse au chauffage des bitumes industriels

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Masseverlustes von Industriebitumen nach Erwärmung

This European Standard was approved by CEN on 10 February 2009.

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## Foreword

This document (EN 13303:2009) 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of an identical text or by endorsement, at the latest by September 2009, and conflicting national standards shall be withdrawn at the latest by September 2009.

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 13303:2003.

The explicit design of the aluminium shelf presented on Figure 1 is no more mandatory (for quality control reasons), but only proposed as an example. Moreover, users of this standard are invited to gather comparative information with EN 12607-2, to facilitate the withdrawal of this standard at the next systematic review.

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, 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 loss in mass of industrial bitumen after heating. The method is used to detect volatile components.

NOTE The users of the method are encouraged to gather comparative information on binders using this standard, EN 13303 and EN 12607-2 [1] at 163 °C to facilitate the withdrawal of EN 13303 at the next systematic review.

**WARNING — The use of this standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to 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 1426, *Bitumen and bituminous binders – Determination of needle penetration*

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

3 Terms and definitions

For the purposes of this document, the following term and definition applies.

3.1  
**loss on heating**  
ratio in between the loss in mass after heating a sample and its initial mass, expressed as a percentage of the latter

4 Principle

A weighed sample is heated for a specified time at a specified temperature and is re-weighed at the conclusion of the heating period.

5 Apparatus

5.1 **Oven**, electrically heated and conforming to the performance requirements for ovens ventilated by natural convection and for operating temperatures up to 180 °C.

The oven shall be rectangular with minimum interior dimensions of 330 mm in each direction. The oven shall have in front a tightly fitting hinged door, which shall provide a clear opening, substantially the same as the interior height and width of the oven. The door may contain a window with dimensions of at least 100 mm x 100 mm, and with two sheets of glass separated by an air space, through which a vertical thermometer (5.3) located as specified in 7.8, may be read without opening the door, or the oven may be provided with an inner glass door through which the thermometer may be observed on opening the outer door momentarily.

The oven shall be adequately ventilated by convection currents of air and for this purpose shall be provided with openings for the entrance of ambient air and egress of heated air and vapours. These openings may be of any size and arrangement provided the temperature requirements of the test are met.

## **5.2 Rotating shelf (see Figure 1a and 1b)**

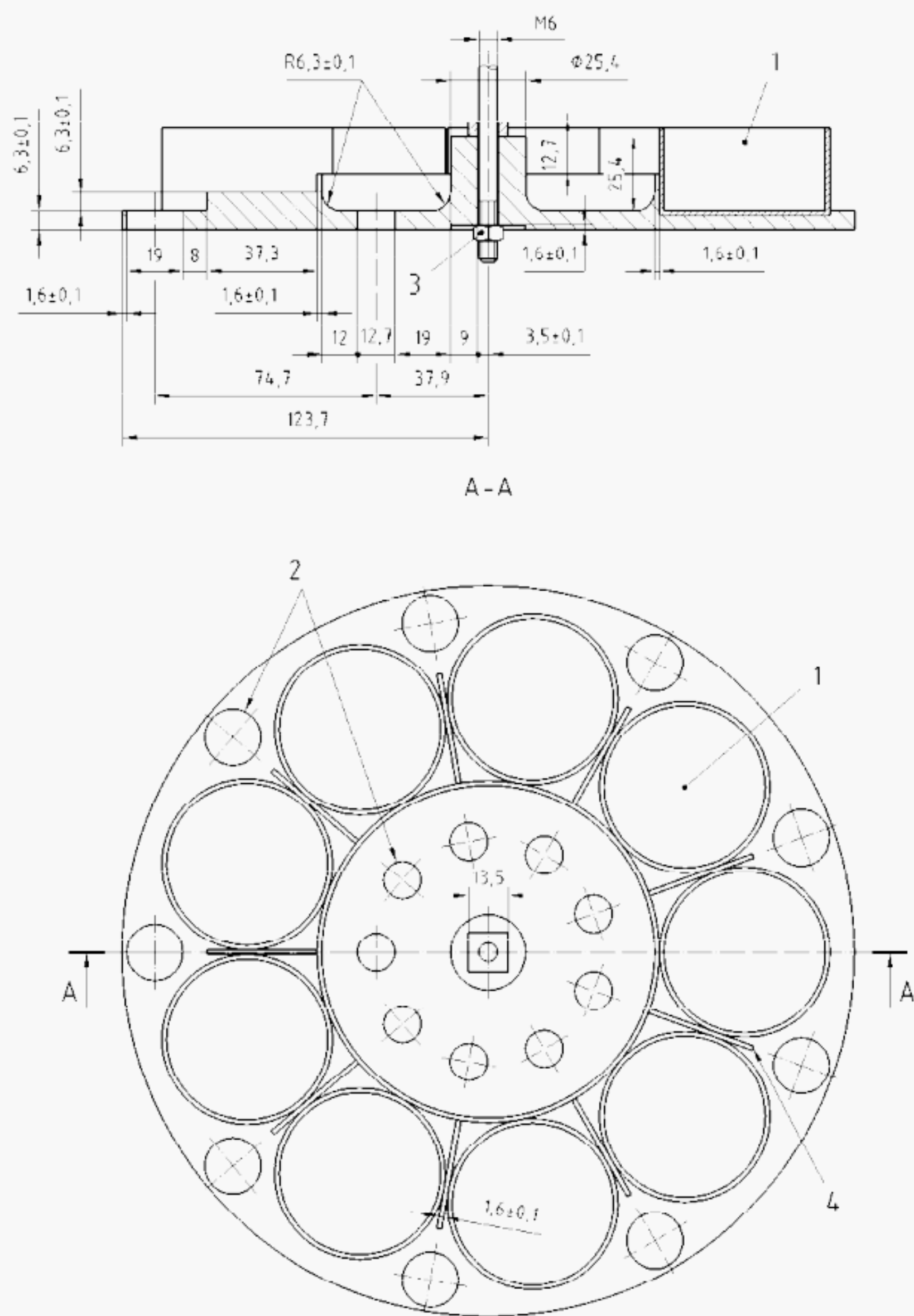
The oven shall be provided with a circular metal shelf having a minimum nominal diameter of 250 mm. The shelf shall be suspended by a vertical shaft and centred with respect to the horizontal interior dimensions. The shelf shall be provided with a mechanical means of rotating it at the rate of 5 rpm to 6 rpm. The shelf shall be vertically located as close to the centre of the oven as permitted by compliance with the requirements of the procedure regarding thermometer placement.

NOTE An example of the shelf is shown in Figure 1a and 1b.



Dimensions in millimetres

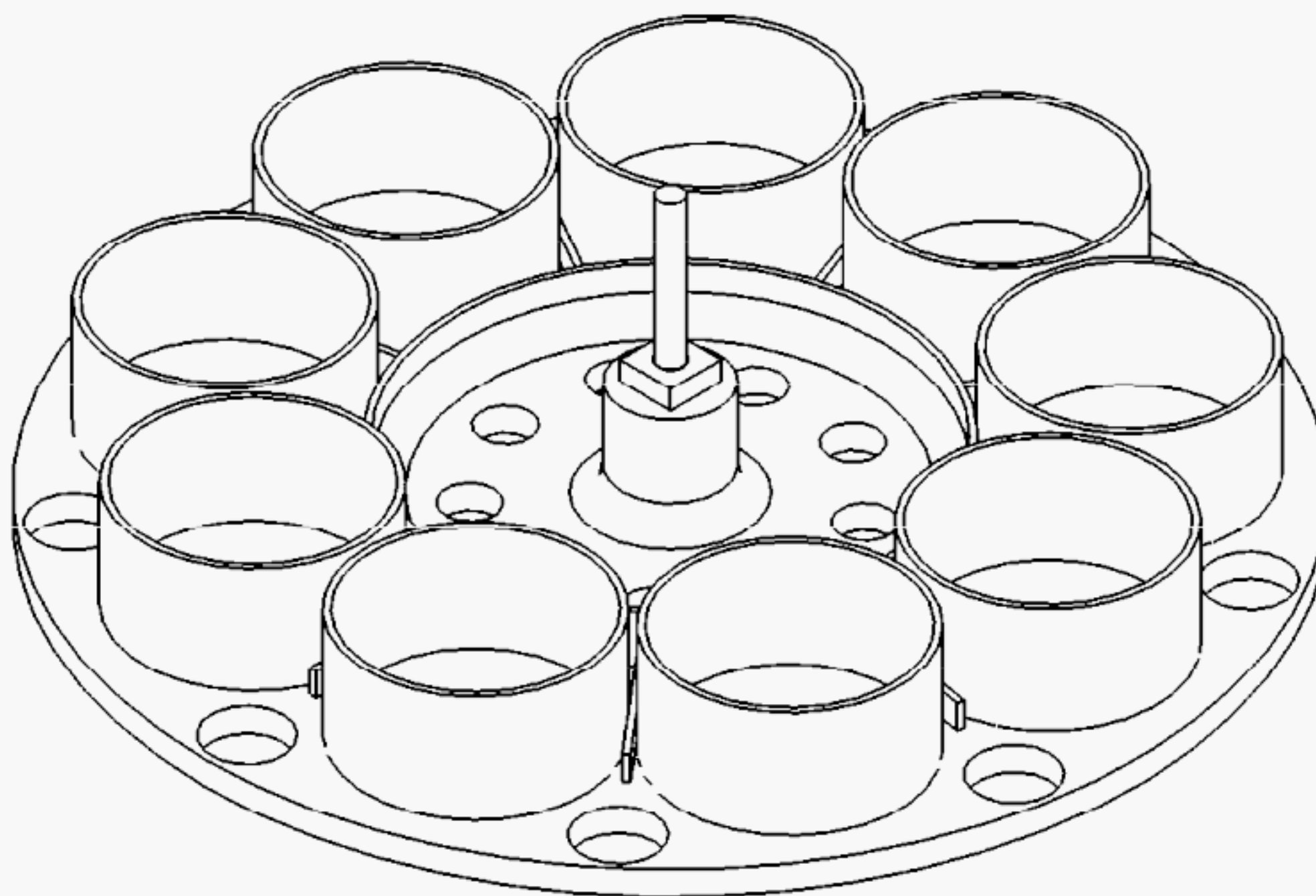
Tolerances not mentioned on the figure are equal to 0,5 mm



Key

- 1. Position of boxes
- 2. 9 holes and ribs spaced equally
- 3. Nut of 6

Figure 1a — Aluminium shelf (example)



**Figure 1b — 3D representation of the aluminium shelf (example)**

**5.3 Thermometer**, in accordance with Annex A.

Other temperature 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.

**5.4 Test sample container**, metal or glass, cylindrical in shape, and with a flat bottom.

The inside dimensions shall be approximately: diameter, 55 mm and depth, 35 mm i.e. conforming to the dimensions of the container specified in EN 1426 for penetration lower than 160 x 0,1 mm.

**5.5 Balance**, with a reading accuracy of 0,01 g.

## 6 Sampling

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

## 7 Procedure

**7.1** Weigh the container (5.4) and record the mass,  $m_c$ , to the nearest 0,01 g reading.

**7.2** Place  $50,0 \text{ g} \pm 0,5 \text{ g}$  of the sample of the material to be tested in the container. Weigh the container and the sample, and record the mass,  $m_1$ , to the nearest 0,01 reading. Carry out the test on duplicate samples.

**7.3** If the sample has been heated to facilitate its transfer, cool the container and sample to ambient temperature before making the initial weighing (see sub-clause 7.2).



- 7.4 Bring the oven to a temperature of 163 °C.
- 7.5 Place the container with the weighed sample on the circular shelf into one of the recesses.
- 7.6 Close the oven and rotate the shelf for the duration of the test at a rate of 5 rpm to 6 rpm.
- 7.7 Maintain the oven temperature at  $(163 \pm 1)$  °C for 5 h after the samples have been introduced. The 5 h period commences when the temperature reaches 162 °C, and ensure that, in no case, the total time that a sample is in the oven exceeds 5 h and 15 min.
- 7.8 Determine the oven temperature by means of the specified thermometer (5.3), which is placed in a vertical position approximately 19 mm inside the perimeter of the shelf, and with the bottom of the thermometer bulb 6 mm above the shelf. If an electrical thermometer is used it should be placed parallel to the shaft in the same position as before but not rotated.
- 7.9 At the conclusion of the heating period, remove the sample from the oven.
- 7.10 Cool the sample to room temperature under a cover protecting it from dust (e.g. dessiccator).
- 7.11 Weigh the container and the sample. Record the mass,  $m_2$ , to the nearest 0,01 g reading.
- 7.12 It is permissible to carry out multiple tests simultaneously.

8 Calculation

The loss in mass after heating of the test sample is expressed as a percentage, calculated by means of the following equation:

$$\text{Loss in mass after heating} = 100 \times \frac{m_1 - m_2}{m_1 - m_c} \tag{1}$$

where

- $m_c$  is the mass of the container, in grams (see sub-clause 7.1);
- $m_1$  is the mass of the container and sample before heating, in grams (see sub-clause 7.2);
- $m_2$  is the mass of the container and sample after heating, in grams (see sub-clause 7.11).

9 Expression of results

Express the result as the mean of two results, in percentage.

10 Precision

10.1 Repeatability

The difference between two successive 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, differ by more than the value given in Table 1 in only one case in twenty.



10.2 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, differ by more than the value given in Table 1 in only one case in twenty.

Table 1 — Precision values

Loss in mass	Repeatability r	Reproducibility R
< 0,5 %	0,1	0,2
≥ 0,5 % to < 1,0 %	0,2	0,4
≥1,0 % to < 2,0 %	0,3	0,6
≥ 2,0 %	15 % of mean	30 % of mean

11 Test report

The test report shall contain at least the following information:

- a) type and complete identification of the sample under test;
- b) reference to this European Standard;
- c) result obtained (see Clause 9);
- d) any unusual behaviour (e.g. foaming);
- e) any deviation, by agreement or otherwise, from the procedure specified;
- f) date of the test.

Annex A  
(normative)

Specifications of thermometer

Temperature range	155 °C to 170 °C
Immersion	total
Scale marks:	
Subdivisions	0,5 °C
Long lines at each	1 °C and slightly longer at 5 °C
Numbers at each	5 °C
Maximum line width	0,15 mm
Maximum scale error	0,5 °C
Expansion chamber permits heating to	200 °C
Total length	150 mm to 160 mm
Stem outside diameter	5,5 mm to 7,0 mm
Bulb length	10 mm to 15 mm
Bulb outside diameter	5,0 mm min but not greater than stem
Scale location	
Distance between bottom of bulb and line at 155 °C	50 mm to 60 mm
Length of scale range	40 mm to 60 mm

NOTE      The thermometer ASTM 13C / IP 47C has been found suitable



## Bibliography

- [1] EN 12607-2, *Bitumen and bituminous binders – Determination of the resistance to hardening under the influence of heat and air – Part 2: TFOT method*