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

National foreword

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The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum Testing and Technology.

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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 1 May 2017.

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Contents

European foreword.....	3
1 Scope	4
2 Normative references	4
3 Terms and definitions	4
4 Principle	4
5 Apparatus.....	4
6 Sampling.....	7
7 Procedure.....	8
8 Calculation.....	8
9 Expression of results.....	9
10 Precision.....	9
10.1 Repeatability.....	9
10.2 Reproducibility	9
11 Test report.....	9
Annex A (informative) Characteristics of thermometer for the reference temperature (163 °C)	10
Bibliography.....	11

European foreword

This document (EN 13303:2017) has been prepared by Technical Committee CEN/TC 330 "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of an identical text or by endorsement, at the latest by March 2018, and conflicting national standards shall be withdrawn at the latest by March 2018.

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

This document supersedes EN 13303:2009.

The primary objective of this revision has been to delete the mercury containing thermometer as reference. Other changes are:

- The method can be performed at various test temperatures, but 163 °C is the reference temperature.
- Clause 9 Expression of results has been made more precise.

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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. The method can be performed at various test temperatures, but 163 °C is the reference temperature.

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 transition to the use of only one standard.

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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 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.

The reference point for the temperature is specified in 7.8.

NOTE If the oven is equipped with a thermometer that requires visual reading, the door may contain a window with dimensions of at least 100 mm × 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.

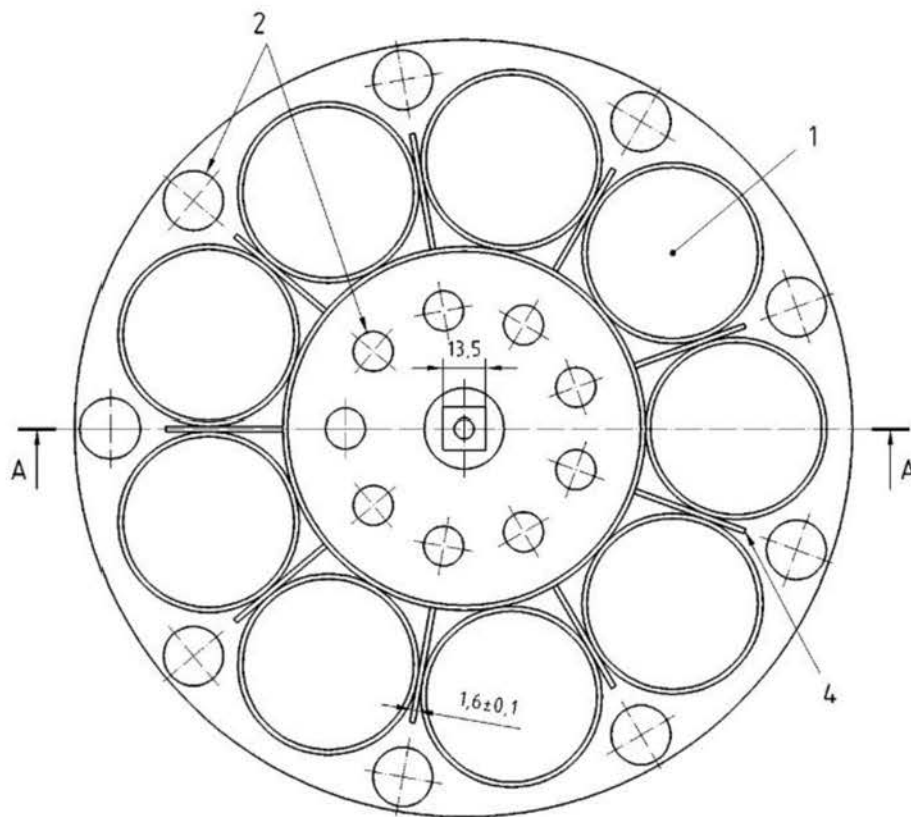
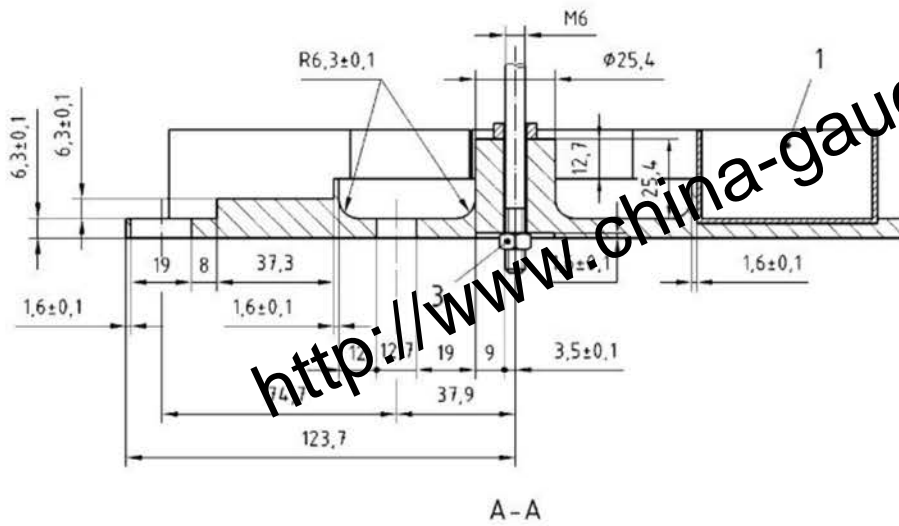
5.2 Rotating shelf (see Figures 1 and 2)

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 r/min to 6 r/min. 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 placement of the sensor of the temperature measuring device.

NOTE An example of the shelf is shown in Figures 1 and 2.

Dimensions in millimetres

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



Key

- 1 Position of boxes
- 2 9 × 2 holes spaced equally
- 3 Nut of 6
- 4 9 ribs spaced equally

Figure 1 — Aluminium shelf (example)



Figure 2 — 3D representation of the aluminium shelf (example)

5.3 Temperature measuring device.

A temperature measuring device (combining sensor and reading unit) shall

- have a range from at least 155 °C to 170 °C,
- be readable to 0,1 °C or less and,
- have an accuracy of 0,5 °C or better.

Sensors based on platinum resistance thermometers have been found suitable but other principles are also allowed. The thermal response time of the sensor shall be comparable with the former used reference (see informative Annex A). The temperature measuring device shall be calibrated regularly.

A solid stem mercury thermometer (which used to be the former reference thermometer as described in Annex A) is also allowed if national regulations permit its use.

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 × 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 $(50,0 \pm 0,5)$ 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 g 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 7.2).
- 7.4 Bring the oven to the test temperature.
- 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 r/min to 6 r/min.
- 7.7 Maintain the oven temperature at the test temperature ± 1 °C for 5 h after the samples have been introduced. The 5 h period commences when the oven temperature reaches 1 °C below the test temperature, 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 temperature measuring device (5.3). The temperature sensor shall be placed in a fixed vertical position approximately 19 mm inside the perimeter of the shelf, and with the bottom of the sensor 6 mm above the shelf. If solid stem mercury thermometer is used (see informative Annex A) it can be mounted on an arm to the shaft parallel with it in the same position as before to allow the thermometer to rotate with the shelf during the heating period.
- 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. desiccator).
- 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:

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

where

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

9 Expression of results

The results of percentage loss in mass after heating for two containers are considered valid if the difference of the two measurements is equal to or less than 0,05 % mass fraction absolute.

Express the percentage of loss in mass after heating as the average of a minimum of two valid determinations to the nearest 0,01 % mass fraction absolute.

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) test temperature
- e) any unusual behaviour (e.g. foaming);
- f) any deviation, by agreement or otherwise, from the procedure specified;
- g) date of the test.

Annex A
(informative)

Characteristics of thermometer for the reference temperature (163 °C)

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 AS/TM 13C / IP 47C has been found suitable

Bibliography

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

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