

English Version

Bitumen and bituminous binders - Determination of the elastic recovery of modified bitumen

Bitumes et liants bitumineux - Détermination du retour élastique des bitumes modifiés

Bitumen und bitumenhaltige Bindemittel - Bestimmung der elastischen Rückstellung von modifiziertem Bitumen

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Contents

Page

| | |
|------------------------------------|---|
| Foreword..... | 3 |
| 1 Scope | 4 |
| 2 Normative references | 4 |
| 3 Terms and definitions | 4 |
| 4 Principle | 4 |
| 5 Apparatus | 5 |
| 6 Preparation of test samples..... | 6 |
| 7 Procedure | 7 |
| 8 Calculation..... | 7 |
| 9 Expression of results | 8 |
| 10 Precision..... | 8 |
| 11 Other test conditions..... | 8 |
| 12 Test report | 8 |
| Bibliography | 9 |

Foreword

This document (EN 13398:2010) 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of an identical text or by endorsement, at the latest by November 2010, and conflicting national standards shall be withdrawn at the latest by November 2010.

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 13398:2003.

This document contains three significant changes compared to EN 13398:2003:

- 1) The test temperature is no longer only 25 °C, other temperatures are admissible to perform the test.
- 2) Dimensioning of the sample and keeping this sample at the specified test temperature are now in line with EN 13589.
- 3) The formula for calculating the elastic recovery is now expressed by mentioning the elongation L , which also enables to determine the elastic recovery in case of premature .break (i.e. due to brittleness).

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 elastic recovery of bituminous binders in a ductilometer at the test temperature (typically 25 °C or 10 °C; other temperatures can be used).

It is especially applicable to bituminous binders modified with thermoplastic elastomers, but can also be used with other bituminous binders which generate only small recovery.

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 13589, *Bitumen and bituminous binders — Determination of the tensile properties of modified bitumen by the force ductility method*

ISO 5725 (all parts), *Accuracy (trueness and precision) of measurement methods and results*

3 Terms and definitions

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

3.1

bitumen thread

test specimen of moulded bitumen, stretched to a thread

3.2

half-threads

two pieces obtained, when a bitumen specimen has been stretched by 200 mm to a thread and then cut in the middle

3.3

elastic recovery

expressed as a percentage of the distance between the ends of the half-threads, which has developed 30 min after the division relative to the elongation length of 200 mm

4 Principle

A bituminous binder specimen is stretched at the test temperature and a constant rate of 50 mm/min to a predetermined elongation (200 mm). The bitumen thread thus produced is cut in the middle to obtain two halves of thread. After a predetermined time for recovery has elapsed, the shortening of the half threads is measured and expressed as the percentage of the elongation length.

5 Apparatus

Usual laboratory apparatus and glassware, together with the following:

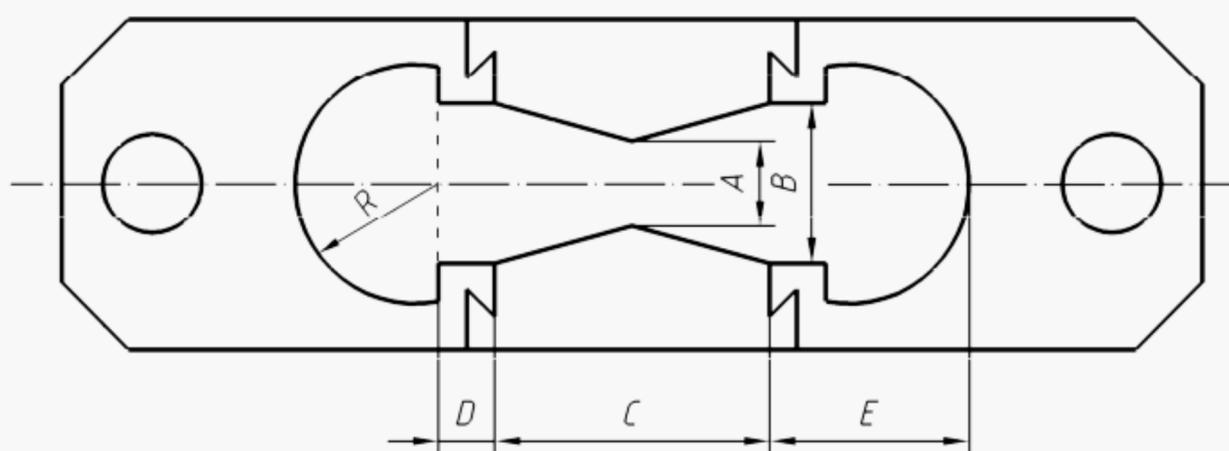
5.1 Specimen moulding equipment.

The moulds shall be made of metal, shall consist of two halves, and shall have the dimensions given in Figure 1.

The ends for the moulds are known as clips and are similar to those specified in EN 13589. The inner radius R of the clips should be of $(15,5 \pm 0,7)$ mm, the opening width B ($20,0 \pm 0,2$) mm and the inner length of the clips $E (= R + D)$ should be $(22,8 \pm 0,9)$ mm (see Figure 1).

IMPORTANT — Tolerances of dimensions D and R are larger than usual, thus ASTM D113 mould can also be used.

Both halves of a mould shall be kept in place by two diametrically opposed sliding pins. The moulds shall be placed on a base plate, also made of metal, and pressed together by the knurled screw or by a plate whilst the test specimens are being cast.



Key

| | Dimension mm | Tolerance mm |
|-------------|-----------------|-----------------|
| A | 10,0 | $\pm 0,2$ |
| B | 20,0 | $\pm 0,2$ |
| C | 30,0 | $\pm 0,3$ |
| D | 7,3 | $\pm 0,5$ |
| $E = R + D$ | 22,8 | $\pm 0,9$ |
| R | 15,5 | $\pm 0,7$ |
| Thickness | 10,0 | $\pm 0,1$ |

Figure 1 — Mould type (normative dimensions, informative design)

5.2 Ductilometer, consisting of a water bath (5.2.1) with a temperature control (5.2.3) and a traction device (5.2.2).

5.2.1 Water bath

The water bath shall allow elongation of the test specimen to at least 200 mm. It shall be designed in such a way that at least two specimens can be tested in parallel. The distance between each mould and between the walls of the water bath shall be at least 10 mm. The water level shall be such that there is at least 25 mm of water above and below the specimen.

Circulation of the bath water via a thermostat and, possibly, additional thermal insulation of the water bath shall guarantee the required test temperature within $\pm 0,5$ °C. Circulation shall be maintained during the test at a reduced rate of approximately 1,5 l/min.

NOTE It may be advisable to direct the water stream at the inlet against a baffle plate to avoid turbulent water flow.

5.2.2 Traction device

The traction device shall allow two specimens to be tested in parallel. The traction plates shall be exactly positioned by a stop switch allowing easy introduction of the pins into the holes of the clips with ease. The drive of the traction device shall be designed in such a way that the combined movement of the pins during the entire test is steady at a constant rate of $(50,0 \pm 2,5)$ mm/min. The drive should be sufficiently powerful to overcome high deformation resistance at the beginning of the test without speed loss.

5.2.3 Temperature control

The temperature control shall be capable of maintaining the test temperature within the water bath of the ductilometer constant within a limit of $\pm 0,5$ °C.

5.3 Knife, with a straight blade of at least 40 mm length or a plane blade.

5.4 Mould release agent, mixture of one part glycerine and one part dextrine or with silicone..

5.5 Scissors

5.6 Ruler with scale divisions to 1 mm.

6 Preparation of test samples

6.1 General

Ensure that the laboratory sample is representative of the bituminous binder to be analysed, in accordance with EN 58. Ensure that the laboratory sample is homogeneous and non-contaminated. The test samples shall be prepared in accordance with EN 12594.

6.2 Preparation of the moulds

The base plate and the inner walls of the sides-pieces shall be applied with a thin coat of the release agent. Assemble the clips and sides on the base plate and arrest them with the knurled screw. Ensure that the specified distance between the sides is achieved (Dimension A on Figure 1).

6.3 Filling the moulds

The slightly heated moulds shall be carefully filled with the bitumen up to the surfaces of the moulds with a meniscus protruding.

6.4 Keeping specimens at specified temperature

Keep the moulded specimens for about 1 h at room temperature then remove the excess sample using a heated knife. Reject any specimens exhibiting defects. Place the moulded specimens in the water bath maintained at the test temperature for (90 ± 10) min, before testing.

The time from filling the moulds to the start of stretching shall be kept within (150 ± 10) min.

6.5 Temperature check of the water bath

Check the temperature of the water at the far end of each traction plate by the pins. The temperature shall not differ by more than $\pm 0,5$ °C from the specified test temperature.

7 Procedure

Once the level filled moulds have been kept at the test temperature for 90 min, remove the moulds from the base plate and the sides of the moulds and transfer the bitumen specimens to the traction plates. Then stretch the specimens at the test temperature $\pm 0,5$ °C and at a speed of $(50,0 \pm 2,5)$ mm/min up to an elongation of (200 ± 1) mm. Within 10 s after the traction device is halted, cut the bitumen threads in the middle with a pair of scissors, produce two half-threads. Thirty minutes after cutting the bitumen threads, use a ruler to measure the lengths between the ends of the half-threads and express them in millimetres.

NOTE The operator is allowed to move the half threads slightly in case they are not facing each other properly ("curled ends").

If the thread of one specimen breaks before the elongation of 200 mm and it is not due to a deficient specimen, the laboratory can use the specimen for calculation of elastic recovery under these conditions which shall be stated in the test report. The laboratory shall document their procedure for these cases with respect to the design and operation of the ductilometer.

8 Calculation

For each specimen, calculate the elastic recovery, R_E , as a percentage (absolute), rounded up to 1 % using the following equation:

$$R_E = \frac{d}{L} \times 100 \quad (1)$$

Where

d is the distance between half-threads, in millimetres.

L stretching length. Normally 200 mm is the elongation when the tread is cut. In case of premature break (due to brittleness) L is the length at break.

If the values of elastic recovery determined for both test pieces do not differ by more than 5 % in absolute value, determine the arithmetic mean of these two values.

Otherwise, determine the elastic recovery of an additional test piece. Then calculate the arithmetic mean of the two values which differs the least. However, if their difference differs by more than 5 % in absolute value, ignore the three values and repeat the test with two new test specimens.

9 Expression of results

Report the elastic recovery in percentage rounded to full percent (absolute value).

The arithmetic mean shall be rounded up to 1 % in accordance with ISO 5725 (all parts).

10 Precision

NOTE A Round Robin test carried out according to ISO 5725 (all parts), with 25 European laboratories participating, permitted to establish precision values, which are the best estimation currently known. The following precision data are proposed until results of further round robin tests are available.

10.1 Repeatability

The difference between two single and independent results, obtained by the same operator working 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 4 % 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 7 % in only one case in twenty.

11 Other test conditions

Although this procedure is typically performed at a test temperature of 25 °C or 10 °C, it may be useful to perform it at different temperatures, especially for soft or highly modified binders. If the sample breaks before reaching 200 mm, the length at break and its elastic recovery shall be measured and then the results can be considered valid.

12 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) test temperature;
- d) stretching length (irrespective whether the sample is cut at 200 mm elongation or if the sample breaks before reaching 200 mm elongation);
- e) results of the test (see Clause 9);
- f) any deviation, by agreement or otherwise, from the procedure specified;
- g) date of the test.

Bibliography

- [1] ASTM D113 – 07, *Standard Test Method for Ductility of Bituminous Materials*