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Bitumen and bituminous binders — Determination of the fracture toughness temperature by a three point bending test on a notched specimen

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National foreword

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The UK participation in its preparation was entrusted by Technical Com mittee B/510, Road materials, to Subcommittee B/510/19, Bitumen and related products.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

Bitumen and bituminous binders - Determination of the fracture toughness temperature by a three point bending test on a notched specimen

Bitumes et liants bitumineux - Détermination de la température de résistance à la fissuration par un essai de flexion 3 points sur un barreau entaillé

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Bruchwiderstandstemperatur mittels eines Drei-Punkt-Biegeversuches an einem gekerbten Probekörper

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Foreword

This document (CEN/TS 15963:2014) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR.

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1 Scope

This Technical Specification specifies a method for the determination of the Fracture Toughness temperature, T_{FT}, of bituminous binders by means of a three point bending test on a notched binder sample.

WARNING — The use of this Technical Specification can involve hazardous materials, operations and equipment. This Technical Specification does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this Technical Specification to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use. For environmental reasons, it is recommended to limit the use of products, solvents and energy to minimum in order to reduce the emissions to air, water and soil.

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 12594, *Bitumen and bituminous binders - Preparation of test samples*

3 Terms and definitions

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

3.1
maximum force
F
highest force measured during the bending test

Note 1 to entry: The test is done in the brittle state or close to that, so that the maximum force is considered as the onset of the crack propagation.

Note 2 to entry: Force is expressed in newtons (N).

3.2
displacement at maximum force
D
bending of the test beam from the beginning of the test (from the zero point) to the break point

Note 1 to entry: Displacement is expressed in millimetres (mm).

3.3
work
W
area under the force-displacement curve from the beginning of the test to the break of the sample, i.e. at the maximum force

Note 1 to entry: Work is expressed in newtons·metres (N·m) or in Joules (J).

3.4
Fracture Toughness temperature
T_{FT}
temperature at which the displacement at the maximum force is 0,3 mm (from the zero point)

Note 1 to entry: Fracture Toughness temperature is expressed in degrees Celsius (°C).

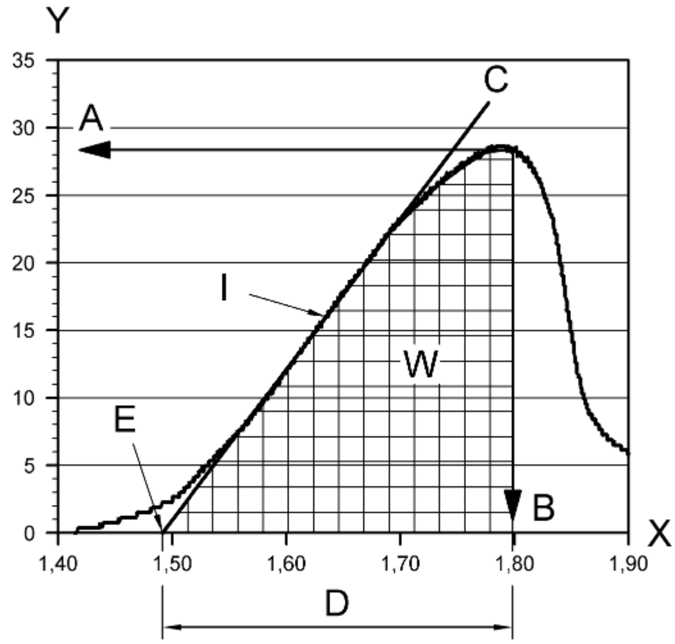
3.5
initial stiffness
S
value calculated as the slope of the tangent of the force-displacement curve at the inflection point

Note 1 to entry: See Figure 1.

Note 2 to entry: Stiffness is expressed in newtons per millimetre (N/mm).

3.6
zero point
intersection of the x-axis and the tangent of the force-displacement curve at the inflection point expressed in millimetres (mm)

Figure 1 presents an example of the force versus displacement curve at a temperature T close to T_{FT}.



Key

X displacement, in millimetres (mm)

Y force, in newtons (N)

A maximum force (defined as *F)*

B X point at maximum force

C tangent at the inflection point

D displacement at maximum force, (*D* = B - E*)*

E zero point

W work as defined in 3.3

I inflection point of *F* versus displacement

Figure 1 — Example of a Force versus displacement curve at temperature T (close to T_{FT})

4 Principle

The notched three point bending test is used to measure cracking performance of unmodified and modified bituminous binder samples. The test sample is a beam with a notch in the middle of one side of the beam. The sample is conditioned in a temperature controlled bath. The beam is placed on two supports with the notch facing downwards and a vertical downward force is applied on the middle of the upper face of the sample. The beam is loaded until failure with a specified displacement rate, whereby force is recorded versus displacement.

NOTE 1 The fracture properties of bituminous binders are strongly dependent on test temperature, loading rate and sample preparation method.

NOTE 2 This test is based on the work done by S. Hesp for the Ministry of Transportation in Ontario, Canada [1].

5 Apparatus

5.1 Testing apparatus

5.1.1 Tension-compression device

A universal tension-compression device, which is capable of sustaining a constant, predetermined displacement rate.

5.1.2 Bending rig

A bending rig consisting of two cylindrical specimen supports, a cylindrical-nosed shaft to apply the load to the mid-point of the test specimen and a load cell mounted in line with the loading shaft.

The dimensions and tolerances of the loading frame are given in Table 1.

5.1.3 Loading system

A loading system, which is capable of applying a rate of displacement of 0,01 mm/s. The specified displacement rate shall fluctuate by no more than ± 10 % over time. The maximum stroke of the instrument shall be at least 20 mm for this test.

5.1.4 Loading shaft

A loading shaft, which is continuous and in line with the load cell and deflection measuring transducer. The T-shape shaft shall have a cylindrical-shaped loading pin in the end. The diameter of the pin shall be (10,0 ± 0,1) mm.

5.1.5 Load cell

A load cell having a minimum capacity of no less than 500 N and an accuracy of ± 1 % above 5 N with a minimum resolution of at least 100 m·N is required. The load cell shall allow the measurement of the force at any time during the test.

5.1.6 LVD-transducer

An LVD transducer or other suitable device to measure the deflection of the sample is necessary. It shall have a linear range of at least 5 mm with an accuracy of 2 µm. The deflection may be measured using this separate transducer or by measuring the vertical movement of the tension/compression device.

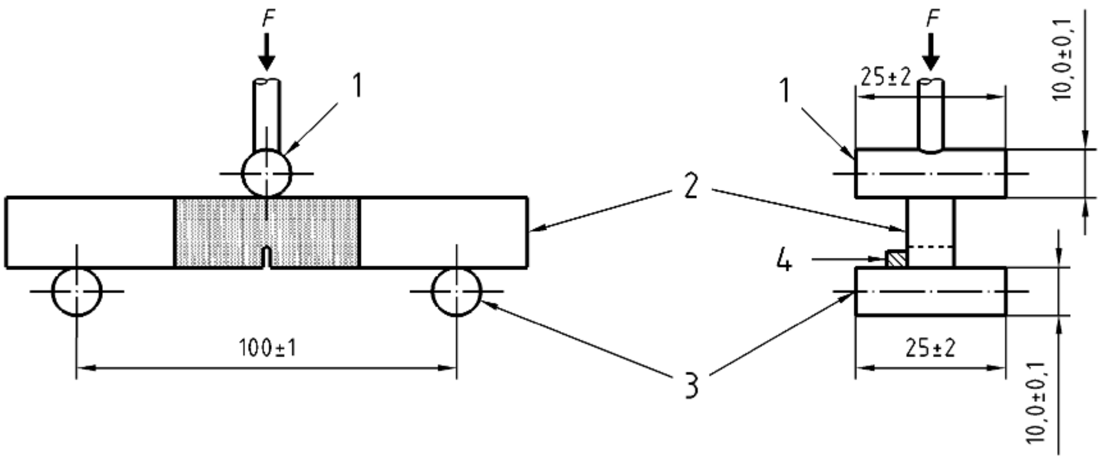
5.1.7 Specimen supports

Specimen supports which shall consist of two non-corrosive metal half-rounds or cylinders with a diameter of (10,0 ± 0,1) mm that are spaced (100 ± 1) mm apart (cylinder centre point to cylinder centre point). The specimen shall be placed perpendicular to the supports and the loading pin. To facilitate that, two vertical alignment pins of 2 mm to 4 mm in diameter can be provided at the back of each support. A schematic diagram of the sample supports is shown in Figure 2.

5.1.8 Ventilated oven, capable of maintaining a temperature up to 200 °C, with an accuracy of ± 1 °C.

The specified temperature shall only be assessed in the surroundings of the sample (see 6.3).

Dimensions in millimetres



Key

1 cylindrical loading shaft pin
2 specimen (see details on Figure 3)
3 specimen supports
4 alignment pin
F force applied

Figure 2 — Bending rig

Table 1 — Measurement and tolerances of the rig

Dimension	Size, mm	Tolerance, mm
Loading shaft pin length	25	±2
Loading shaft diameter	10,0	±0,1
Specimen support length	25	±2
Specimen support diameter	10,0	±0,1
Specimen support span	100	±1

5.1.9 Temperature measurement device

A calibrated temperature transducer capable of measuring the temperature with the accuracy of ± 0,1 °C over the range of - 40,0 °C to 0,0 °C. The temperature measuring head shall be mounted in the near vicinity of the specimen, at a distance of not more than 25 mm from the middle of the specimen.

5.1.10 Liquid bath

A liquid bath shall be capable of maintaining the required test temperature near the test specimen within ± 0,2 °C during the isothermal conditioning and the test procedure, enabling to reach the lower temperature required for testing the specimen.

Bath liquid shall not affect the properties of the bituminous binder being tested.

NOTE 95 % volume ethanol or a 40 % to 50 % mass potassium acetate-water solution has been found to be suitable as a bath liquid.

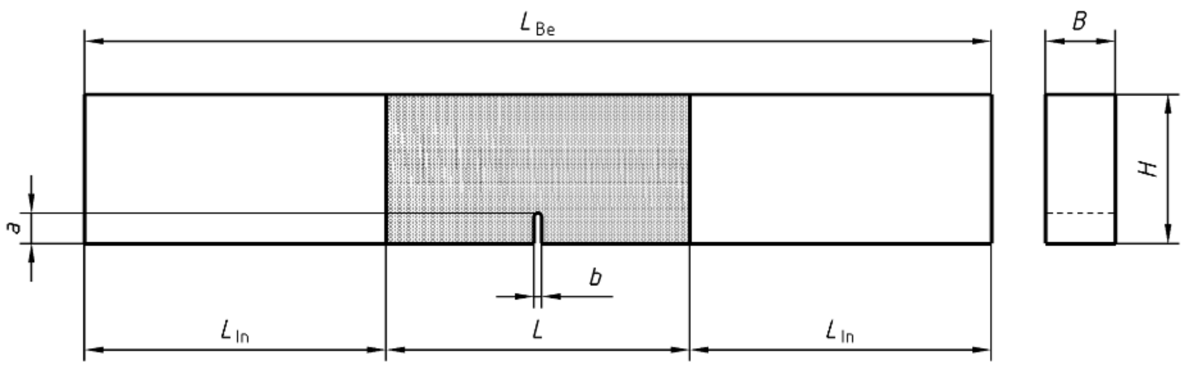
5.1.11 Data acquisition

The data acquisition system shall resolve loads to the nearest 100 mN, record specimen deflection to the nearest 0,5 µm, record time to the nearest 0,1 s and temperature around the sample to the nearest 0,1 °C. The software shall control the measuring system and record time, load, deflection and temperature during the test. Sampling rate of force and displacement should be at least 0,1 s^{-1} (10 Hz).

5.2 Test specimen

A test specimen as shown on Figure 3 is used. The sample is moulded from the bituminous binder to be assessed. Two aluminium inserts are installed at the ends of the mould. The notch is made by installing two sheets of silicone paper into the pre-notched mould frame.

NOTE Silicone paper with a weight of 135 g/m^2 has been found suitable.



Key

L_{Be} beam length

L specimen length

L_{In} insert length

H specimen height

B specimen thickness

a notch depth

b notch thickness

Figure 3 — Specimen geometry

Table 2 —Test specimen dimensions and tolerances

Dimension	Size, mm	Tolerance [a], mm
Beam length, L_{Be}	120,0	±0,3
Insert length, L_{In}	40,0	±0,1
Specimen length, L	40,0	±0,2
Specimen height, H	25,0	±0,5
Specimen thickness, B	12,5	±0,3
Notch depth, a	5,0	±0,1
Notch thickness, b	2 × 25 µm	-

[a] Tolerance is related to the mould.

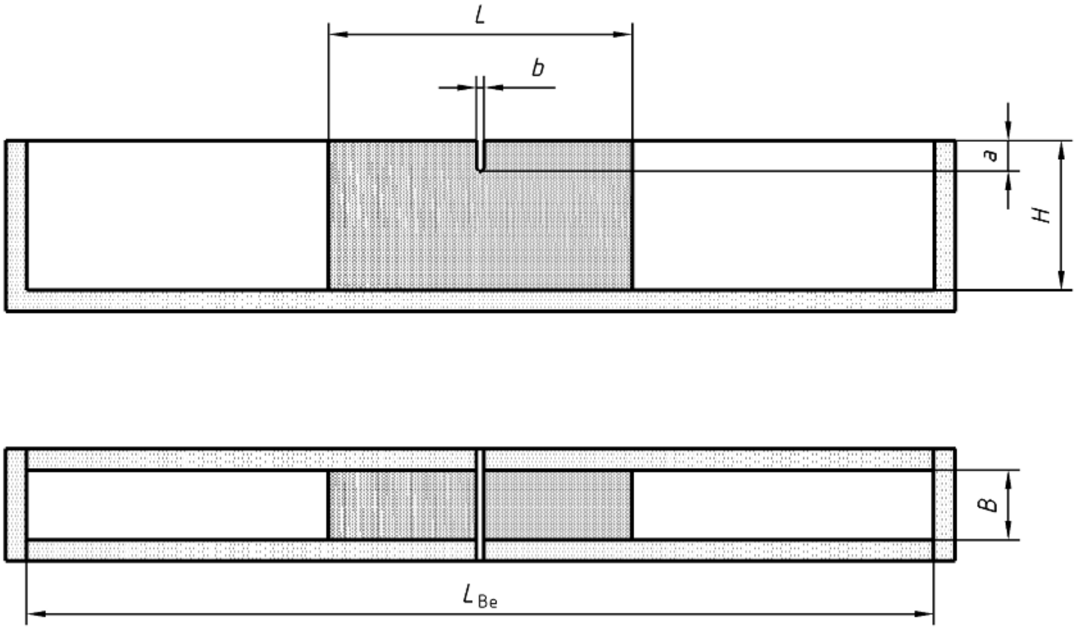
6 Preparation of test samples

6.1 Preparation of test samples

The laboratory sample shall be taken in accordance with EN 58 and the test samples shall be prepared in accordance with EN 12594.

6.2 Preparation of moulds

The moulds for making the samples shall be assembled from aluminium side spacers. On the side spacers and on the bottom a plastic film is glued with an anti-sticking agent to prevent sticking of the bitumen binder to the mould. Two aluminium inserts are installed in the ends of the mould. The surfaces of the aluminium inserts that are in contact with bitumen should be roughened to ensure good adhesion. A series of such assemblies may be clamped together to make more than one specimen at a time. Figure 4 shows the different parts of the mould. Figure 5 shows a picture of such an example assembly and its parts.



Key

L_{Be} beam length

L specimen length

H specimen height

B specimen thickness

a notch depth

b notch thickness

Figure 4 — Mould assembly for specimen preparation

The mould assembly should be sufficiently tight that no bitumen can flow from the cavity into the spaces between the different parts.

As anti-sticking agent talcum-glycerine mixture in ratio 1/1 or a suitable commercial anti-slicking grease should be used.

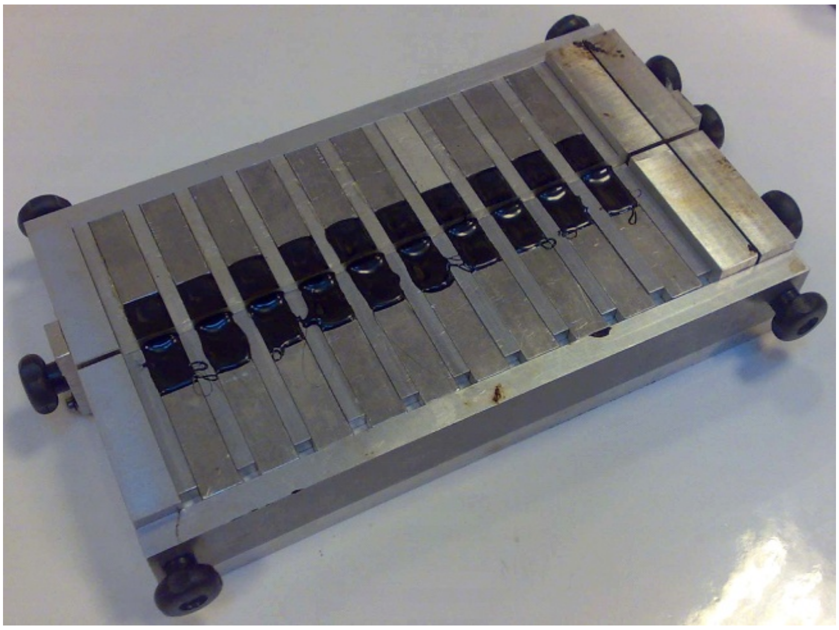


Figure 5 — Photograph of a mould assembly

6.3 Preparation of test specimen

The binder is heated according to EN 12594 to ensure that it readily flows when dispensed from the container. Heat to not more than 100 °C above the expected softening point as defined in EN 1427. For modified bitumen use the same procedure if no other guidance is provided by the supplier. In any case, 200 °C shall not be exceeded. The binder shall be stirred prior to use. Air bubbles trapped in the binder should be avoided because it affects the result.

Install the two sheets of silicone paper tightly into the mould. Install the silicone papers with the silicone sides to each other. The silicone papers shall not be taken away from the specimen after moulding otherwise the slit notch can close down prior to the test.

Heat the mould for 10 min at the sample temperature (see first paragraph above) to ensure easy filling of the mould. It shall be ensured that the silicone papers remain straight, tight and are not glued together with bitumen. If necessary, tighten once again the silicone papers.

The hot binder is poured into the mould. Slightly over-fill the mould to allow for shrinkage during cooling. Let the filled mould cool at the ambient temperature (between 18 °C and 28 °C) for (24 ± 1) h. After cooling, it is not necessary to remove any excess bitumen because this will be the bottom surface of the test sample and this does not affect testing.

Just prior to demoulding, cool the mould containing the test specimens in a cold chamber or a liquid bath to stiffen the test specimens so that they can be readily demoulded without distortion; a temperature of −20 °C during 10 min is recommended. The temperature to which the specimens are brought for demoulding shall never be below the lowest testing temperature.

Testing shall be completed within 4 h after demoulding. If the test is done at several temperatures then it may be necessary to have longer time between demoulding and testing. This may affect the precision of the test. In the meanwhile, between demoulding and conditioning (see 7.2), the demoulded test specimens shall be stored on a flat surface at a temperature that will prevent specimens from creeping. Storage between approximately 2 °C and 7 °C is appropriate (i.e. usual temperature in laboratory fridge).

NOTE 1 Excessive duration of cooling can cause unwanted hardening of the binder and effect on the test result.

NOTE 2 During demoulding, the specimens will be handled with care to prevent distortion, a warped test specimen can affect the measured values.

NOTE 3 After demoulding, specimen dimensions can change due to shrinkage.

NOTE 4 The correct positioning of the beam on the supports of the bending rig is of vital importance. It may be helpful to put markings on the inserts to make the positioning on the supports easier.

7 Procedure to determine the Fracture Toughness temperature T_{FT}

7.1 Number of tests

Fracture tests at a specified temperature shall be repeated a minimum of three times.

7.2 Specimen conditioning

The specimens shall be conditioned at the test temperature in the liquid bath for (60 ± 5) min. The temperature of the liquid bath shall be set within ± 0,2 °C during the conditioning period.

7.3 Loading rate and test temperature

The loading rate shall be kept at 0,01 mm/s and shall vary by no more than ± 10 % during tests.

The temperatures shall cover a range so as to yield both purely brittle failure (complete linearity) as well as a limited degree of ductility (nonlinearity) followed by a failure. At least one temperature should be below and one above the Fracture Toughness temperature. The temperature step is recommended to be maximum 5 °C.

7.4 Validity of the test results

If the displacement is more than 1 mm from the zero point, lower the test temperature. The test results with a 1 mm displacement or less may be used for the final calculation of the Fracture Toughness temperature.

At the end of the test, immediately finish disintegrating the specimen beam along the fracture plane and check the fracture surfaces. The fracture plane shall be perpendicular to the longitudinal axis of the beam. If the loading shaft is not perfectly perpendicular to the specimen, crack turns around the propagation axis and the fracture zone will not be a plane perpendicular to the longitudinal axis. Verify also that the pre-notch is correctly localized and not twisted or curved. These failings may cause a false result, which should be discarded.

7.5 Fracture Toughness temperature, T_{FT}

Fracture Toughness temperature is considered as the lowest temperature at which this displacement at maximum force is 0,3 mm.

8 Calculation

8.1 Test sample

For each test specimen, the data acquisition software shall record the applied displacement of the test specimen versus the measured force and the loading time. A force-displacement curve as shown on Figure 1 is drawn. The zero point is sought by drawing the tangent at the inflection point of the curve (Figure 1). The software shall calculate values as follows or a manual evaluation is made in the same fashion:

— maximum force, *F*;

— displacement at maximum force, *D*;

— initial stiffness, *S*;

— work done until maximum force, *W*.

8.2 Results and temperature

8.2.1 General

The following values shall be calculated for each test temperature *T*. Three replicates per temperature shall be performed. And at least two test temperatures are required.

8.2.2 Maximum force, *F*

Maximum force, *F*, at the test temperature *T*, is calculated as the arithmetic mean of at least three successful measurements expressed in newtons (N), with two significant figures.

8.2.3 Work, *W*

Work, *W*, at a test temperature *T*, is calculated as the arithmetic mean of at least three successful measurements expressed in newtons·metres (N·m), with two significant figures.

8.2.4 Displacement at maximum force, *D*

Displacement at maximum force, *D*, at the test temperature *T*, is calculated as the arithmetic mean of at least three successful measurements. The resulting value is expressed in millimetres (mm) with three significant figures.

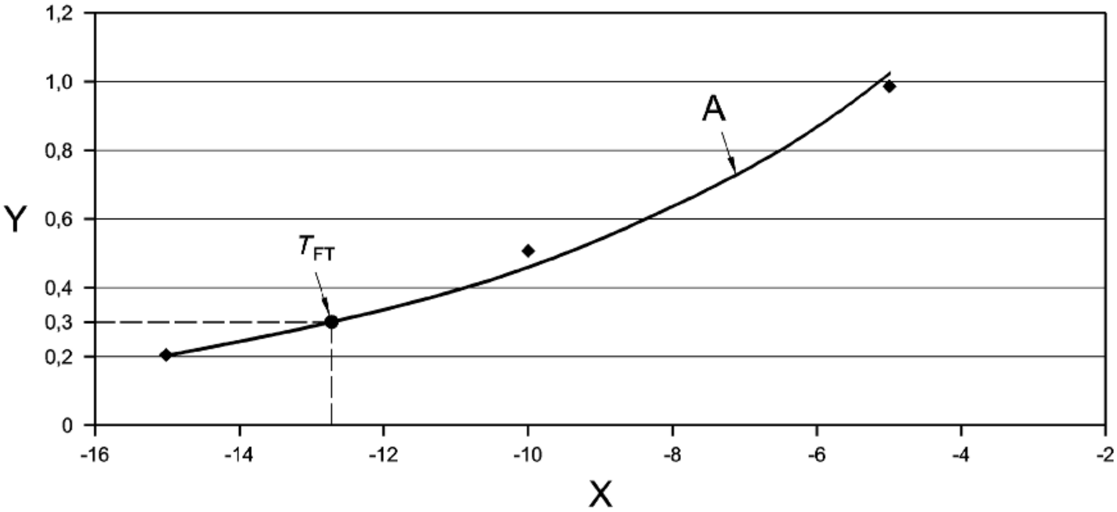
8.2.5 Initial stiffness, *S*

Initial stiffness, *S*, at the test temperature *T*, is calculated as the arithmetic mean of at least three successful measurements. The resulting value is expressed in newtons per millimetre (N/mm), with three significant figures.

8.3 Fracture Toughness temperature, T_{FT}

The Fracture Toughness temperature T_{FT} is the lowest temperature where the displacement at the maximum force is 0,3 mm. T_{FT} is expressed in degrees Celsius (°C), rounded to the nearest integer.

The Fracture Toughness temperature, T_{FT}, is sought by fitting an exponential function to the temperature versus displacement data points and calculating the temperature corresponding to the displacement of 0,3 mm (see Figure 6). A linear interpolation may be used if two temperatures are available on both sides of the Fracture Toughness temperature. Test temperatures that give a displacement at the maximum force less than 1 mm are allowed to be used in the final T_{FT} calculation.



Key

X temperature, in degrees Celsius (°C)

Y displacement at the maximum force, in millimetres (mm)

A fitted exponential curve from the experimental measurements

T_{FT} Fracture Toughness temperature

Figure 6 — Example of displacement at maximum force versus *T*

9 Expression of results

9.1 Fracture Toughness temperature, T_{FT}

The Fracture Toughness temperature, T_{FT} is expressed in degrees Celsius (°C), rounded to the nearest integer.

9.2 Other properties

The other calculated properties, maximum force, work, displacement at maximum force and initial stiffness, at test temperatures should be expressed, if especially asked for.

10 Precision

NOTE Precision values have been determined through a round robin test.

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, exceed the values given in Table 3 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, exceed the values given in Table 3 in only one case in twenty.

Table 3 — Estimated repeatability and reproducibility

	Repeatability, r, °C	Reproducibility, R, °C
Fracture Toughness temperature, $T_{FT,}$ (°C)	3	6

11 Test report

The test report shall contain at least the following information:

a) testing laboratory;

b) type and identification of the sample under test;

c) a reference to this Technical Specification, i.e. CEN/TS 15963;

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

e) date of the test;

f) results of the test (see Clause 9).

Annex A
(informative)

Calculation of Critical Stress Intensity factor

A.1 General

These calculations apply only when mechanical material behaviour can be considered as elastic. In a first approximation, these calculations can be applied when force-displacement curve is linear.

A.2 Terms and definitions

A.2.1 Critical stress intensity factor, K_{IC}

The critical stress intensity factor, K_{IC}, refers to a measure of material strength in the brittle state in the presence of a sharp notch. (See ASTM D5045-96 [2], ASTM E399-90 [3], ASTM E616-89 [4] and ASTM E1290-93 [5] for further details.)

It has been demonstrated that the geometry used in this Technical Specification (bitumen + aluminium) is comparable to a homogeneous sample for the stress intensity calculation [6].

A.2.2 Definitions

F	Load applied onto the specimen	N
F_f	Maximum loading force	N
W	Work up to failure	N·m
H	Specimen height	m
B	Specimen thickness	m
a	Notch depth	m
α	Notch versus height ratio ($\alpha = a/H$)	-
L_S	Specimen support span	m

A.3 Calculations

A.3.1 Critical stress intensity factor, K_{IC}

Critical stress intensity factor at the brittle state for this geometry is determined in the brittle state (linear-elastic behaviour) from the failure load according to the following relationship:

$$K_{IC} = \left(\frac{F_f}{B \times \sqrt{H}}\right)\frac{3}{2} \times \left(\frac{L_S}{H}\right) \times \frac{\sqrt{\alpha}}{(1+2\alpha) \times (1-\alpha)^{3/2}}\left[1{,}99 - \alpha(1-\alpha)(2{,}5 - 3{,}93\alpha + 2{,}7\alpha^2)\right]$$

With the geometry used in this Technical Specification:

$$K_{IC} = F_f \times 2\,377 \text{ N/m}^{3/2} \text{ for } \alpha = a/H = 0{,}2$$

where

K_{IC} is the fracture toughness or critical stress intensity factor in $N/m^{3/2}$;

F_f is the failure force in newtons (N).

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