BS EN 12697-4:2015

BSI Standards Publication

Bituminous mixtures — Test methods

Part 4: Bitumen recovery: Fractionating column

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National foreword

This British Standard is the UK implementation of EN 12697-4:2015. It supersedes BS EN 12697-4:2005 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee B/510/1, Asphalt products.

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Foreword

This document (EN 12697-4:2015) has been prepared by Technical Committee CEN/TC 227 “Road materials”, the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by December 2015 and conflicting national standards shall be withdrawn at the latest by December 2015.

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 12697-4:2005.

The following is a list of changes since the previous edition:

— the series title no longer makes the method exclusively for hot mix asphalt;

— definitions for precision have been removed;

— Formula (1) in 5.2.1 was updated;

— 5.3.3 has been extended;

— Figures 2 and 3 have been corrected;

— the Figure for CO_2 supply has been added.

This European Standard is one of a series of standards as follows:

— EN 12697-1, *Bituminous mixtures — Test methods for hot mix asphalt — Part 1: Soluble binder content*

— EN 12697-2, *Bituminous mixtures — Test method — Part 2: Determination of particle size distribution*

— EN 12697-3, *Bituminous mixtures — Test methods for hot mix asphalt — Part 3: Bitumen recovery: Rotary evaporator*

— EN 12697-4, *Bituminous mixtures — Test methods — Part 4: Bitumen recovery: Fractionating column* [the present document]

— EN 12697-5, *Bituminous mixtures — Test methods for hot mix asphalt — Part 5: Determination of the maximum density*

— EN 12697-6, *Bituminous mixtures — Test methods for hot mix asphalt — Part 6: Determination of bulk density of bituminous specimens*

— EN 12697-7, *Bituminous mixtures — Test methods for hot mix asphalt — Part 7: Determination of bulk density of bituminous specimens by gamma rays*

— EN 12697-8, *Bituminous mixtures — Test methods for hot mix asphalt — Part 8: Determination of void characteristics of bituminous specimens*

— EN 12697-10, *Bituminous mixtures —Test methods for hot mix asphalt — Part 10: Compactability*

— EN 12697-11, *Bituminous mixtures — Test methods for hot mix asphalt — Part 11: Determination of the affinity between aggregate and bitumen*

— EN 12697-12, *Bituminous mixtures — Test methods for hot mix asphalt — Part 12: Determination of the water sensitivity of bituminous specimens*

— EN 12697-13, *Bituminous mixtures — Test methods for hot mix asphalt — Part 13: Temperature measurement*

— EN 12697-14, *Bituminous mixtures — Test methods for hot mix asphalt — Part 14: Water content*

— EN 12697-15, *Bituminous mixtures — Test methods for hot mix asphalt — Part 15: Determination of the segregation sensitivity*

— EN 12697-16, *Bituminous mixtures — Test methods for hot mix asphalt — Part 16: Abrasion by studded tyres*

— EN 12697-17, *Bituminous mixtures — Test methods for hot mix asphalt — Part 17: Particle loss of porous asphalt specimen*

— EN 12697-18, *Bituminous mixtures — Test methods for hot mix asphalt — Part 18: Binder drainage*

— EN 12697-19, *Bituminous mixtures — Test methods for hot mix asphalt — Part 19: Permeability of specimen*

— EN 12697-20, *Bituminous mixtures — Test methods for hot mix asphalt — Part 20: Indentation using cube or cylindrical specimens (CY)*

— EN 12697-21, *Bituminous mixtures — Test methods for hot mix asphalt — Part 21: Indentation using plate specimens*

— EN 12697-22, *Bituminous mixtures — Test methods for hot mix asphalt — Part 22: Wheel tracking*

— EN 12697-23, *Bituminous mixtures — Test methods for hot mix asphalt — Part 23: Determination of the indirect tensile strength of bituminous specimens*

— EN 12697-24, *Bituminous mixtures — Test methods for hot mix asphalt — Part 24: Resistance to fatigue*

— EN 12697-25, *Bituminous mixtures — Test methods for hot mix asphalt — Part 25: Cyclic compression test*

— EN 12697-26, *Bituminous mixtures — Test methods for hot mix asphalt — Part 26: Stiffness*

— EN 12697-27, *Bituminous mixtures — Test methods for hot mix asphalt — Part 27: Sampling*

— EN 12697-28, *Bituminous mixtures — Test methods for hot mix asphalt — Part 28: Preparation of samples for determining binder content, water content and grading*

— EN 12697-29, *Bituminous mixtures — Test method for hot mix asphalt — Part 29: Determination of the dimensions of a bituminous specimen*

— EN 12697-30, *Bituminous mixtures — Test methods for hot mix asphalt — Part 30: Specimen preparation by impact compactor*

— EN 12697-31, *Bituminous mixtures — Test methods for hot mix asphalt — Part 31: Specimen preparation by gyratory compactor*

— EN 12697-32, *Bituminous mixtures — Test methods for hot mix asphalt — Part 32: Laboratory compaction of bituminous mixtures by vibratory compactor*

— EN 12697-33, *Bituminous mixtures — Test methods for hot mix asphalt — Part 33: Specimen prepared by roller compactor*

— EN 12697-34, *Bituminous mixtures — Test methods for hot mix asphalt — Part 34: Marshall test*

— EN 12697-35, *Bituminous mixtures —Test methods for hot mix asphalt — Part 35: Laboratory mixing*

— EN 12697-36, *Bituminous mixtures — Test methods for hot mix asphalt — Part 36: Determination of the thickness of a bituminous pavement*

— EN 12697-37, *Bituminous mixtures — Test methods for hot mix asphalt — Part 37: Hot sand test for the adhesivity of binder on precoated chippings for HRA*

— EN 12697-38, *Bituminous mixtures — Test methods for hot mix asphalt — Part 38: Common equipment and calibration*

— EN 12697-39, *Bituminous mixtures — Test methods for hot mix asphalt — Part 39: Binder content by ignition*

— EN 12697-40, *Bituminous mixtures — Test methods for hot mix asphalt — Part 40: In situ drainability*

— EN 12697-41, *Bituminous mixtures — Test methods for hot mix asphalt — Part 41: Resistance to de-icing fluids*

— EN 12697-42, *Bituminous mixtures — Test methods for hot mix asphalt — Part 42: Amount of foreign matter in reclaimed asphalt*

— EN 12697-43, *Bituminous mixtures — Test methods for hot mix asphalt — Part 43: Resistance to fuel*

— EN 12697-44, *Bituminous mixtures — Test methods for hot mix asphalt — Part 44: Crack propagation by semi-circular bending test*

— EN 12697-45, *Bituminous mixtures — Test methods for hot mix asphalt — Part 45: Saturation Ageing Tensile Stiffness (SATS) conditioning test*

— EN 12697-46, *Bituminous mixtures — Test methods for hot mix asphalt — Part 46: Low temperature cracking and properties by uniaxial tension tests*

— EN 12697-47, *Bituminous mixtures — Test methods for hot mix asphalt — Part 47: Determination of the ash content of natural asphalts*

— prEN 12697-48, *Bituminous mixtures — Test methods for hot mix asphalt — Part 48: Interlayer Bonding*[1)]

— EN 12697-49, *Bituminous mixtures — Test methods for hot mix asphalt — Part 49: Determination of friction after polishing*

— prCEN/TS 12697-50, *Bituminous mixtures — Test methods — Part 50: Resistance to Scuffing*[1)]

— FprCEN/TS 12697-51, *Bituminous mixtures — Test methods — Part 51: Surface shear strength test*

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1) Currently at Enquiry stage.

— prEN 12697-52, *Bituminous mixtures — Test methods — Part 52: Conditioning to address oxidative ageing*[2)]

— prEN 12697-53, *Bituminous mixtures — Test methods — Part 53: Cohesion increase by spreadability-meter method* [3)]

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2) In preparation.

3) Currently at drafting stage.

Introduction

WARNING — The method described in this European Standard may require the use of dichloromethane (methylene chloride), 1.1.1 trichlorethane, benzene, trichlorethylene, xylene, toluene or other solvent capable of dissolving bitumen. These solvents are hazardous to health and are subject to occupational exposure limits as detailed in relevant legislation and regulations.

Exposure levels are related to both handling procedures and ventilation provision and it is emphasized that adequate training should be given to staff employed in the usage of these substances.

1 Scope

This European Standard specifies a test method for the recovery of soluble bitumen from bituminous mixtures from pavements in a form suitable for further testing. The procedure is suitable for the recovery of paving grade bitumen and is also suitable for mixtures containing volatile matter such as cut-back bitumen but the results may be less precise. This European Standard is the reference method for mixtures containing volatile matter, but the rotary evaporator procedure (see EN 12697-3) for mixtures with paving grade bitumen.

NOTE There is limited experience of recovery when polymer-modified bitumen is used.

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

EN 12697-1:2012, *Bituminous mixtures — Test methods for hot mix asphalt — Part 1: Soluble binder content*

EN 12697-3, *Bituminous mixtures — Test methods for hot mix asphalt — Part 3: Bitumen recovery: Rotary evaporator*

EN 12697-38, *Bituminous mixtures — Test methods for hot mix asphalt — Part 38: Common equipment and calibration*

3 Terms and definitions

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

3.1
soluble binder content
percentage by mass of extractable binder in an anhydrous sample determined by extracting the binder from the sample

Note 1 to entry: Extraction may be followed by binder recovery.

3.2
insoluble binder content
percentage by mass of binder that adheres to the aggregate after extraction

4 Principle

The bitumen is separated from the sample by dissolving in dichloromethane (or other suitable solvent). After removal of undissolved solids, the bitumen solution is concentrated by atmospheric distillation in a fractionating column. The last traces of solvent are removed from the concentrate by distillation at a temperature of 100 °C above the expected softening point or 175 °C, whichever is the higher, with the pressure reduced from atmospheric pressure 100 kPa to 20 kPa and with the aid of a stream of carbon dioxide gas. When cutback bitumens containing very volatile fluxes, e.g. white spirit, are being recovered the carbon dioxide gas is omitted.

5 Apparatus

5.1 Apparatus for the extraction of the soluble bitumen

Suitable container with stopper, in which the sample and solvent can be agitated together, or other apparatus for the extraction of soluble bitumen defined in EN 12697-1.

NOTE The use of the hot extraction methods in EN 12697–1 may harden the binder and hence affect the results from subsequent tests. However, this hardening is usually regarded as approximately balancing the softening resulting from any remaining solvent.

5.2 Apparatus for the clarification of the bitumen solution

For separation of solids from the bitumen solution, a sample-tube centrifuge, a continuous centrifuge or a filtration system may be used.

Centrifuges are suitable for separation of solids from any bitumen solutions and are the recommended apparatus for use with this method. The filtration apparatus may not be suitable for the separation of solids from all types of bituminous solutions but it has been included in this method because of the general availability of this equipment in asphalt testing laboratories. If difficulties are experienced using a pressure filter the centrifuge technique should be used.

5.2.1 Sample tube centrifuge, capable of developing an acceleration of at least 15 000 m/s^2 in accordance with the following formula:

$$a = 1{,}097 \times n^2 \times r \times 10^{-6} \quad (1)$$

where

- a is the acceleration, expressed in metres per second squared (m/s^2);
- n is the number of revolutions, expressed in revolutions per minute (r/min);
- r is the radius to the bottom of the tubes (internal) when rotating, expressed in millimetres (mm).

The centrifuge tubes shall be fitted with effective closures.

The speed of rotation shall be verified regularly in accordance with EN 12697-38 to ensure that the centrifuge maintains its performance at all times. The centrifuge shall be maintained in accordance with EN 12697-3.

NOTE A typical centrifuge of this type, suitable for this method, carries four or six tubes of 200 ml or 500 ml capacity rotating at 3 000 r/min at a radius (as defined above) of 250 mm.

5.2.2 Continuous laboratory centrifuge, that takes a continuous feed of material, giving a continuous discharge of solution and capable of achieving an acceleration of 25 000 m/s^2.

5.2.3 A pressure filter, of an appropriate size.

5.2.4 An air pump, for supplying oil-free air at about 200 kPa.

5.2.5 A supply of filter papers with a minimum retention size of 11 µm, to fit the pressure filter.

NOTE A pressure filter taking a paper of 270 mm diameter is suitable.

5.3 Distillation apparatus (see Figure 1)

5.3.1 500 ml round-bottomed flask of heat resisting glass fitted with a three-necked glass adaptor.

The central neck is used either to accommodate a stirrer (see Figures 2 or 3) or a glass tube from 4 mm to 6 mm internal diameter for sweeping carbon dioxide through the flask when required. A 250 ml stoppered separating funnel is fitted to one side neck of the multiple adapter. The other side neck is connected to the fractionating column followed by an efficient water-cooled glass condenser and receiver system. The fractionating column is of the Dufton type or Vigreux type having an effective length from 300 mm to 400 mm and may be vacuum jacketed. The receiver system includes a tap by which the main receiver can be isolated from the condenser. All connections shall be made by means of ground-glass joints.

5.3.2 Oil bath, suitable for heating the distillation flask and capable of raising the temperature of the oil to 100 °C above the softening point of the recovered bitumen or 175 °C, whichever is the higher, a thermometer for the oil bath and a means for raising and lowering the bath.

5.3.3 Flow meter having a range from 0 ml to 30 ml free flow of carbon dioxide per minute at 15 °C and 20 kPa pressure together with a CO_2 supply tube (Figure 4).

5.3.4 Suitable means of reducing pressure, e.g. a filter pump or electrically operated vacuum pump with a gauge indicating pressures from approximately 10 kPa to 100 kPa.

5.3.5 Thermometer, capable of covering the temperature range from 100 °C to 200 °C with an accuracy of ± 0,5 °C.

6 Solvent and other materials

6.1 Dichloromethane (methylene chloride) or other suitable solvent such as 1.1.1 trichlorethane, benzene, trichlorethylene, xylene or toluene.

6.2 Petroleum jelly or glycerol, to seal glass joints.

6.3 Silica Gel, passing a 63 µm sieve.

6.4 Carbon dioxide, under pressure in cylinders which are fitted with gas regulators.

6.5 Porous pot, to be used as anti-bumping material.

7 Procedure

7.1 Extraction of the bitumen and removal of insoluble matter

7.1.1 Place a sufficient amount of the bituminous mixture to contain sufficient bitumen for performing binder test(s) in a suitable container and add about 1 500 ml of dichloromethane (or other suitable solvent) and sufficient silica gel to absorb any water present in the sample. Agitate the contents of the container until the mineral aggregate is clear and all of the soluble bitumen has dissolved.

7.1.2 Allow the bitumen solution to stand for about 10 min, decant the bitumen solution through a 63 µm sieve and then free from insoluble material. This can be achieved by either a) or b), were a) is the reference method:

a) Separation by centrifuging:

Remove insoluble matter from the bitumen solution by centrifuging at an acceleration of at least 15 000 m/s^2 for (20 ± 5) min if using a sample tube centrifuge or by passing the bitumen solution through a continuous centrifuge. If a continuous centrifuge is used, the minimum acceleration shall be 25 000 m/s^2 and the rate of discharge shall not exceed 150 ml/min.

b) Separation by filtration:

Fit the pressure filter with filter paper. Pass the bitumen solution through the filter paper under pressure not exceeding 200 kPa. Wash the sample until the outflow is almost colourless. Filter aids are not permissible.

If difficulties are experienced in filtering the bitumen solution, the centrifuge technique should be used.

Ash contents to EN 12697–1:2012, C.2, should be carried out occasionally on recovered bitumens to ensure that excessive mineral matter is not present.

7.1.3 During separation of solids from the bitumen solution make every effort to prevent any moisture from entering the bitumen solution. Pay particular attention to reducing any evaporation of the dichloromethane (or other suitable solvent) to a minimum, thereby limiting the risk of the formation of condensation.

7.1.4 Transfer the bitumen solution to a glass container and store it in the dark until the beginning of the bitumen recovery distillation.

7.2 Assembling and checking the apparatus for air leaks

Check the assembled distillation apparatus for air leaks with a carbon dioxide supply and gas delivery tube in position. Use the minimum of petroleum jelly or glycerol to lubricate the joints. Do not use silicone lubricants. Reduce the pressure in the apparatus to about 20 kPa, isolate the apparatus from the source of reduced pressure and test the apparatus for air tightness with a pressure rise of 2 kPa over a period of 10 min or less being considered acceptable.

7.3 Distillation procedure

7.3.1 Replace the gas delivery tube by the stirrer and add anti-bumping material such as porous pot to the flask. Introduce approximately 100 ml of bitumen solution into the distillation flask through the separating funnel and agitate the bitumen solution by the stirrer revolving at about 4 r/s for the glass link stirrer or 2 r/s if the pivoted stirrer is used. Raise the temperature of the oil bath to (100 ± 5) °C. When distillation starts introduce further bitumen solution slowly into the flask, keeping the volume of bitumen solution in the flask at a minimum. In no case shall the volume exceed 250 ml.

7.3.2 When all of the bitumen solution has been added to the flask, allow the contents to concentrate. During concentration allow the temperature of the oil bath to increase gradually with a constant heat input. When the rate of distillation has slackened to about 10 drops per minute increase the temperature of the oil bath over a period from 20 min to 30 min to (100 ± 5) °C above the expected softening point, for bitumens with a softening point above 75 °C, or to (175 ± 5) °C.

If the softening point is not known, use (175 ± 5) °C.

7.3.3 After the rate of distillation has dropped to three or four drops per minute for five consecutive minutes adjust the oil bath so that the oil level is from 10 mm to 20 mm above the liquid level in the flask. Connect the carbon dioxide supply and position the end of the tube at less than 5 mm from the bottom of the flask. Empty the receiver flask.

7.3.4 Pass carbon dioxide through the residue in the flask at 10 ml/min and reduce the pressure in the apparatus gradually over a period from 10 min to 15 min until the pressure has fallen to 20 kPa. During the

reduction of the pressure maintain the flow of carbon dioxide at 10 ml/min by suitable adjustment of the control valve.

7.3.5 Maintain the bath temperature (100 °C above the expected softening point or 175 °C, whichever is the higher), pressure (20 kPa) and flow of carbon dioxide (10 ml/min) for 45 min.

If it is suspected that the bitumen contains a very volatile flux, e.g. white spirit, the use of carbon dioxide should be omitted.

7.3.6 After 45 min isolate the apparatus from the source of reduced pressure and allow the pressure to increase, by the ingress of carbon dioxide at 10 ml/min, until the pressure just reaches, but never exceeds, atmospheric.

7.3.7 Allow the fractionating column to drain and remove the flask. If necessary rotate the flask to mix the contents, especially if any condensed oil is present on the walls of the flask or on the surface of the bitumen. Allow the contents of the flask to cool to a temperature at which they cease to fume, but can be poured, and then transfer them into a suitable container.

7.3.8 In order to avoid the possibility of significant hardening of the bitumen by the dichloromethane (or other suitable solvent), complete the total procedure (extraction and recovery) within 24 h.

8 Preparation of the bitumen for testing

Prepare samples of bitumen in accordance with EN 12594.

9 Test report

The test report of this European Standard shall include at least the following information:

a) reference to this European Standard;

b) name and address of the testing laboratory;

c) a unique serial number for the test report;

d) name of the client;

e) description and an identification of the sample, and the date of receipt;

f) the solvent used;

g) the number of times, if any, that the temperature used for the evaporation rose above 175 °C;

h) the date and time of the test;

i) signature of the person accepting technical responsibility for the test report;

j) that the test has been carried out according to this method.

10 Precision

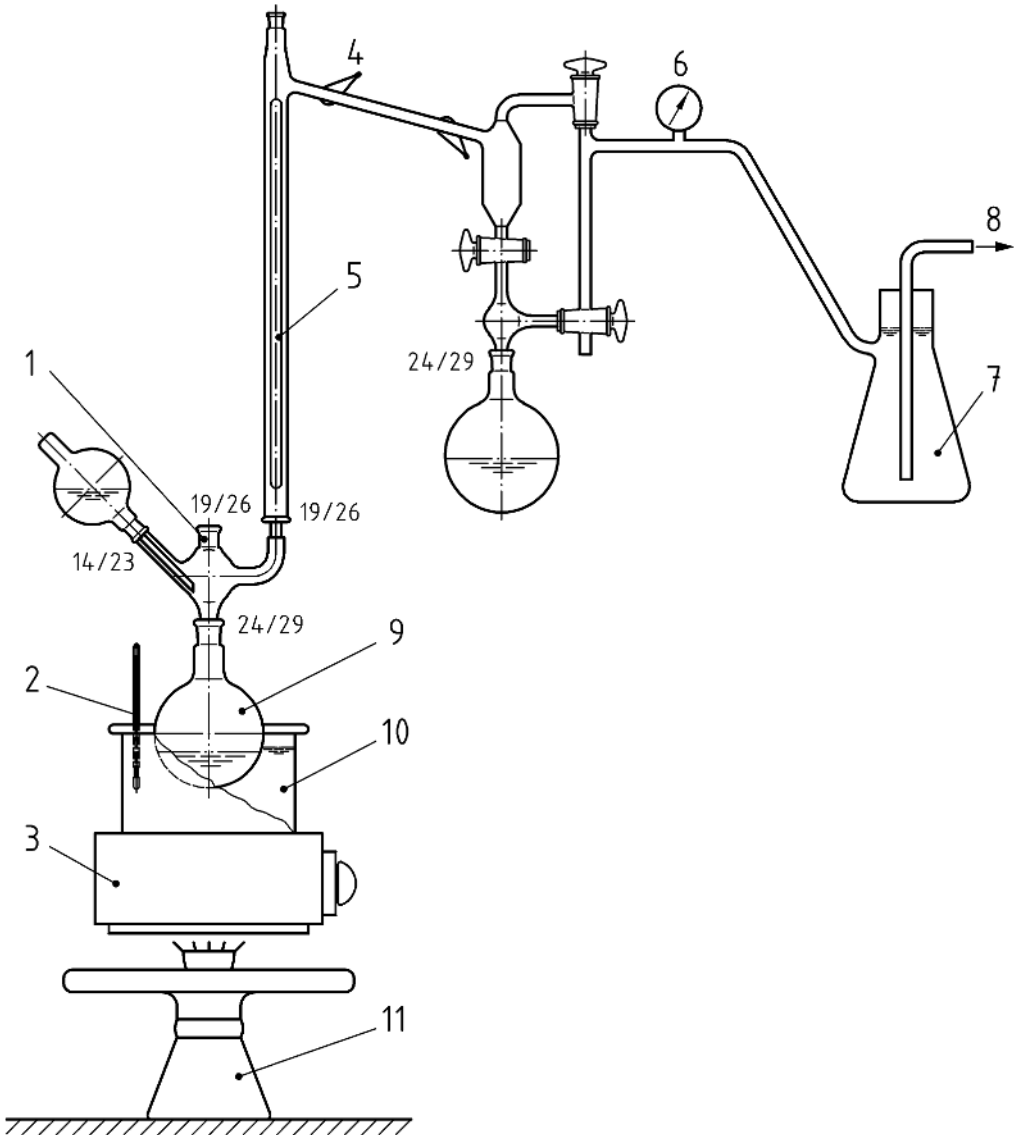
10.1 This method does not produce a result and has no precision. The precision of test methods for penetration and softening point applied to the recovered bitumen is as follows:

Table 1 — Precision values

Method		Repeatability	Reproducibility
Penetration	0,1 mm	5	$1{,}9\sqrt{x}$
Softening point	°C	2,5	3,5
NOTE x is the average of results being compared.			

NOTE The precision exercise was carried using the test procedures described in EN 1426 and EN 1427.

10.2 These precision values have been obtained by statistical examination of inter-laboratory test results and were first published in 1974. They apply to bitumens of up to 120 penetration although indications are they would also apply to bitumens of over 120 penetration.



Key

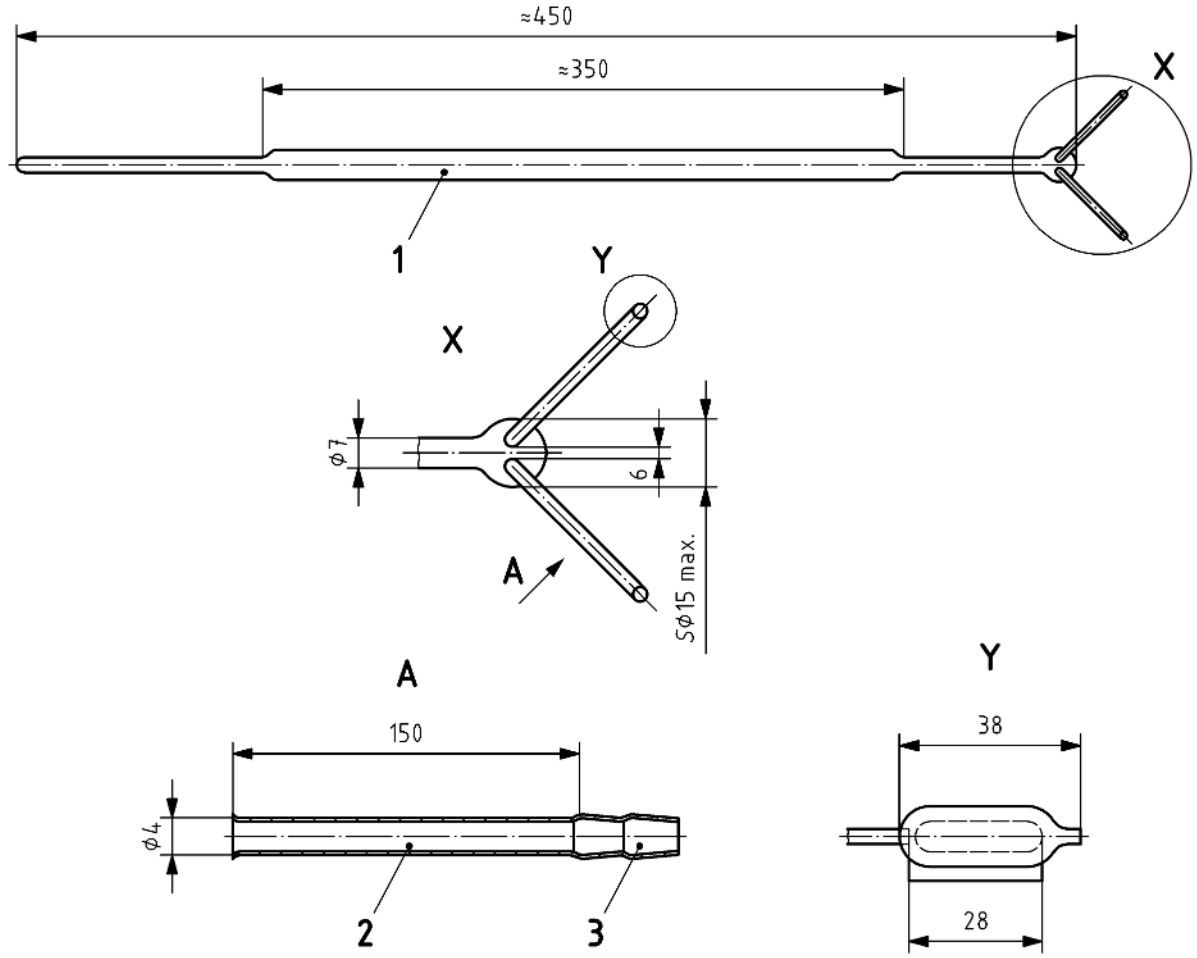
1 cone joint for, separately, fill stirrer and CO_2 tube
2 thermometer with bulb opposite bottom of flask
3 enclosed electrical heater
4 condenser
5 fractionating column
6 vacuum gauge
7 water trap
8 pump
9 500 ml bottom flask
10 oil bath
11 jack

NOTE 1 CO_2 tube reaches to within 5 mm of bottom.

NOTE 2 Internal diameter of glass CO_2 tube 4 mm to 6 mm.

Figure 1 — Distillation apparatus used for the recovery of soluble bitumen

Dimensions in millimetres



Key

1 ground to suit inside diameter of cone joint

2 ground internally

3 19/26 cone

A Support guide for the link stirrer or for the pivoted polytetrafloroethylene stirrer of Figure 3

Figure 2 — Glass link stirrer (all-glass stirrer, 19/26 joint size or equivalent)

Dimensions in millimetres

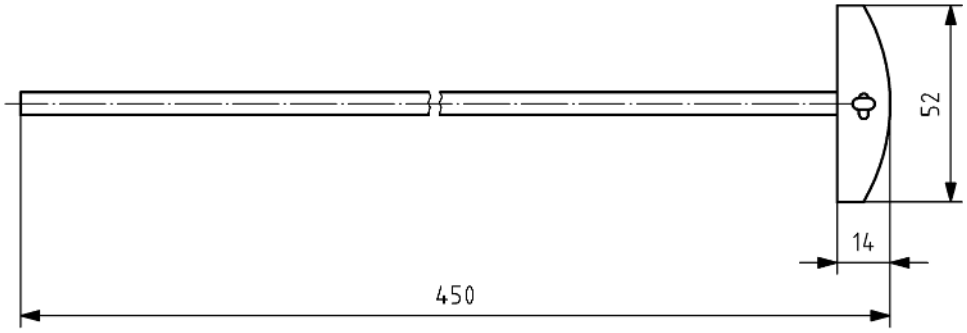


Figure 3 — Pivoted polytetrafluoroethylene blade stirrer

Figure 4 — CO_2 supply tube

Bibliography

[1] EN 1426, *Bitumen and bituminous binders — Determination of needle penetration*

[2] EN 1427, *Bitumen and bituminous binders — Determination of the softening point — Ring and Ball method*

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