BS EN 12697-4:2023

BSI Standards Publication

Bituminous mixtures — Test methods

Part 4: Bitumen recovery: Fractionating column

bsi.

National foreword

This British Standard is the UK implementation of EN 12697-4:2023. It supersedes BS EN 12697-4:2015, which is withdrawn.

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European foreword

This document (EN 12697-4:2023) has been prepared by Technical Committee CEN/TC 227 "Road materials", the secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by October 2023, and conflicting national standards shall be withdrawn at the latest by October 2023.

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 12697-4:2015.

The main changes compared to the previous edition are listed below:

— general update of standard texts according to CEN/CENELEC Internal Regulations Part 3:2019;

— [Clause 1] clarification of the scope;

— [Clause 2] deletion of "for hot mix asphalt" for EN 12697-1 and correction of dated reference;

— [Clause 2] deletion of reference to EN 12697-3. Added to Bibliography;

— [Clause 2] deletion of reference to EN 12697-38;

— [Clause 5.2.1] deletion of paragraph describing calibration and maintenance for the centrifuge with reference to EN 12697-38;

— [Clause 5.3.2] clarification of the temperature capacity for oil bath;

— [Clause 5.3.5] the term "accuracy" for thermometer replaced by "maximum permissible error;

— [Clause 7.1.2] correction of dated reference to EN 12697-1:2020;

— [Clause 7.3.5] completion of tolerances for temperatures;

— [Clause 9] revision of data to be reported;

— [Clause 10.1] completion with reference to Table 1 in paragraph;

— [Figure 2] completion with keys for X and Y.

A list of all parts in the EN 12697 series can be found on the CEN website.

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Introduction

WARNING — The method described in this document 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 document specifies a method for the recovery of soluble bitumen from bituminous mixtures used in road, airfield or similar pavements in a form suitable for further testing.

The method is applicable for the recovery of paving grade bitumen and is the reference method for recovery of soluble bitumen from bituminous mixtures containing volatile matter such as cut-back bitumen.

The method is not applicable for recovery of polymer-modified bitumen.

NOTE EN 12697-3 is the reference method for the recovery of paving grade bitumen and polymer-modified bitumen.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 12594, *Bitumen and bituminous binders - Preparation of test samples*

EN 12697-1:2020, *Bituminous mixtures - Test methods - Part 1: Soluble binder content*

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

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 can 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 cut-back 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 can 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.

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 180 °C 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 a maximum permissible error 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), where 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.

Determination of ash contents according to EN 12697-1:2020, 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 at (100 ± 5) °C above the expected softening point or (175 ± 5) °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 shall include at least the following information:

a) reference to this document;

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

c) the solvent used;

d) any deviations from the procedure;

e) any unusual features observed;

f) the date of the test.

10 Precision

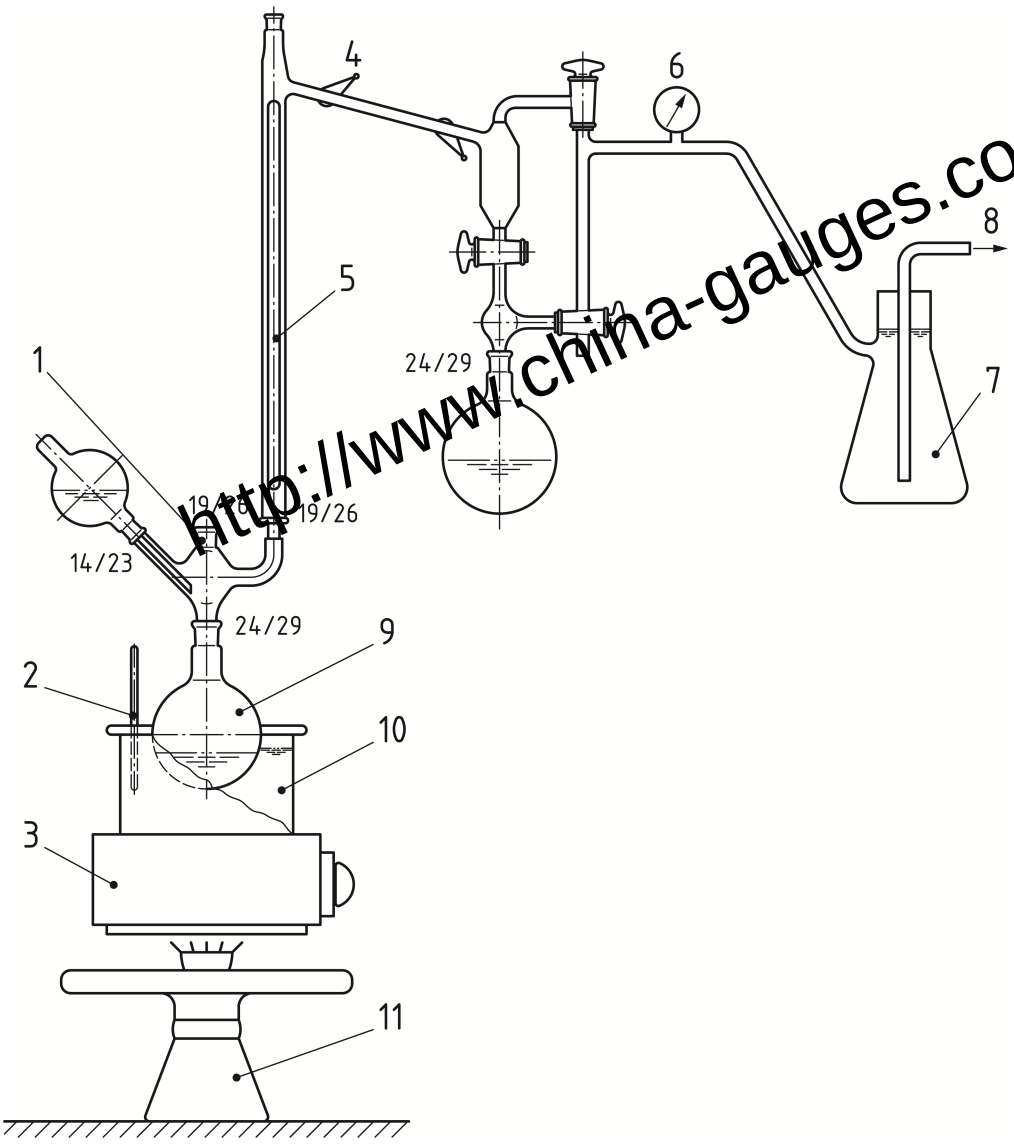
10.1 This method does not produce a result and has no precision. The precision of test methods for penetration and softening point applied to the recovered bitumen is indicated in Table 1.

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 out 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

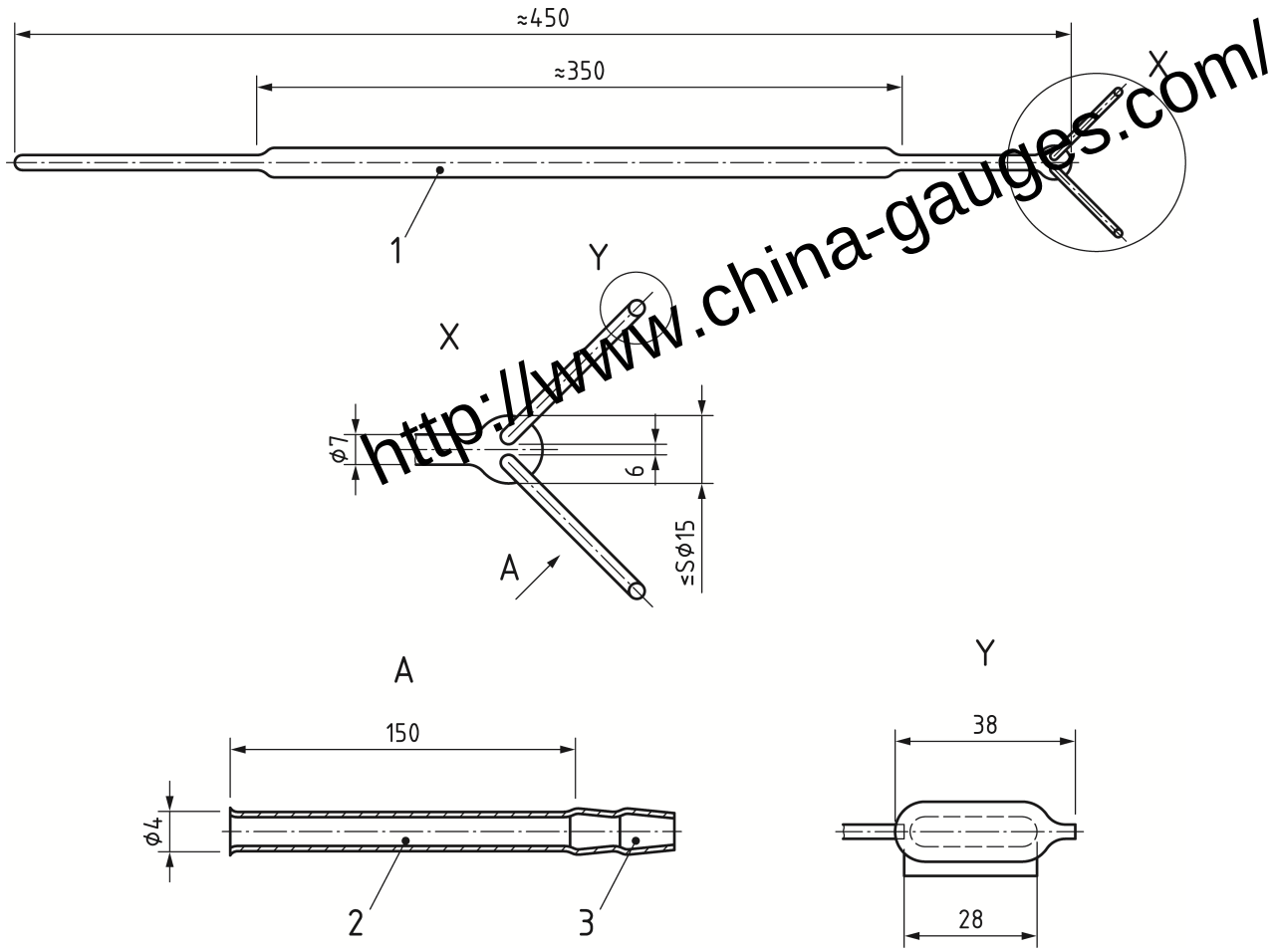
1	cone joint for, separately, fill stirrer and CO_2 tube	7	water trap
2	thermometer with bulb opposite bottom of flask	8	pump
3	enclosed electrical heater	9	500 ml bottom flask
4	condenser	10	oil bath
5	fractionating column	11	jack
6	vacuum gauge		

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	A	Support guide for the link stirrer or for the pivoted polytetrafloroethylene stirrer of Figure 3
2	ground internally	X,Y	enlarged images of glass link stirrer
3	19/26 cone		

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

Dimensions in millimetres

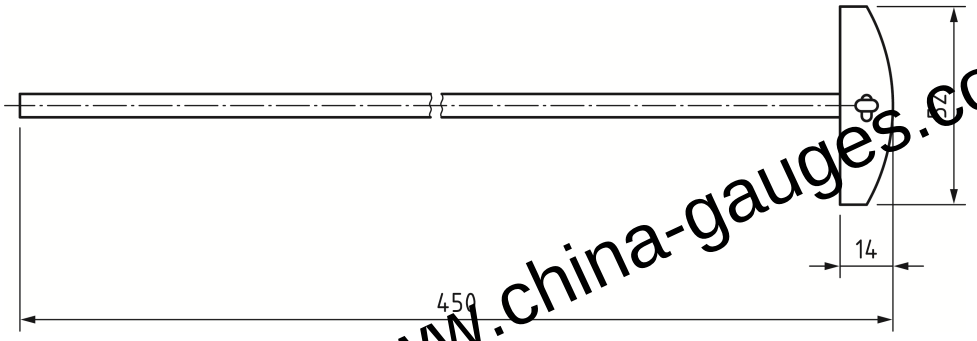


Figure 3 — Pivoted polytetrafluoroethylene blade stirrer

Figure 4 — CO_2 supply tube

Bibliography

[1] EN 12697-3, *Bituminous mixtures - Test methods - Part 3: Bitumen recovery: Rotary evaporator*

[2] EN 1426, *Bitumen and bituminous binders - Determination of needle penetration*

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

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