

BS EN 12697-3:2013



BSI Standards Publication

Bituminous mixtures — Test methods for hot mix asphalt

Part 3: Bitumen recovery: Rotary evaporator

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National foreword

This British Standard is the UK implementation of EN 12697-3:2013. It supersedes BS EN 12697-3:2005 which is withdrawn.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.

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Date	Text affected
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English Version

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

Mélanges Bitumineux - Méthodes d'essai pour enrobés à
chaud - Partie 3: Extraction des bitumes à l'évaporateur
rotatif

Asphalt - Prüfverfahren für Heiasphalt - Teil 3:
Rckgewinnung des Bindemittels: Rotationsverdampfer

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Foreword

This document (EN 12697-3:2013) 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of an identical text or by endorsement, at the latest by November 2013, and conflicting national standards shall be withdrawn at the latest by November 2013.

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 12697-3:2005.

The significant changes from that document include:

- The Scope is clarified.
- The definitions for precision and associated terms have been removed.
- Remaining solvent is redefined as solvent residue.
- The minimum evaporation capacity of solvent has been reduced from 1,0 l/h to 0,85 l/h.
- The container no longer has to be made of glass.
- The warning note about solvent is clarified.
- Silicon grease is permitted as a seal.
- Silica gel has been corrected.
- Pouring of bitumen solution has been edited.
- Ash content reference to EN 12697-1 has been corrected.
- Maximum time for bitumen recovery distillation is reduced.
- Check for air leaks has been removed.
- First phase temperature and second phase pressure with dichloromethane has been changed.
- The minimum final pressure has been made relevant to first phase pressure instead of fixed value for all solvents.
- The use of nitrogen to prevent the bitumen from ageing has been added.
- The note on expected time for distillation has been removed.

This part of EN 12697, *Bituminous mixtures — Test methods for hot mix asphalt*, is one of a series of standards as listed below:

- *Part 1: Soluble binder content*

- *Part 2: Determination of particle size distribution*
- *Part 3: Bitumen recovery: Rotary evaporator*
- *Part 4: Bitumen recovery: Fractionating column*
- *Part 5: Determination of the maximum density*
- *Part 6: Determination of bulk density of bituminous specimens*
- *Part 7: Determination of bulk density of bituminous specimens by gamma rays*
- *Part 8: Determination of void characteristics of bituminous specimens*
- *Part 9: Determination of the reference density*
- *Part 10: Compactibility*
- *Part 11: Determination of the affinity between aggregate and bitumen*
- *Part 12: Determination of the water sensitivity of bituminous specimens*
- *Part 13: Temperature measurement*
- *Part 14: Water content*
- *Part 15: Determination of the segregation sensitivity*
- *Part 16: Abrasion by studded tyres*
- *Part 17: Particle loss of porous asphalt specimen*
- *Part 18: Binder drainage*
- *Part 19: Permeability of specimen*
- *Part 20: Indentation using cube or cylindrical specimens (CY)*
- *Part 21: Indentation using plate specimens*
- *Part 22: Wheel tracking*
- *Part 23: Determination of the indirect tensile strength of bituminous specimens*
- *Part 24: Resistance to fatigue*
- *Part 25: Cyclic compression test*
- *Part 26: Stiffness*
- *Part 27: Sampling*
- *Part 28: Preparation of samples for determining binder content, water content and grading*
- *Part 29: Determination of the dimensions of a bituminous specimen*

- *Part 30: Specimen preparation by impact compactor*
- *Part 31: Specimen preparation by gyratory compactor*
- *Part 32: Laboratory compaction of bituminous mixtures by vibratory compactor*
- *Part 33: Specimen prepared by roller compactor*
- *Part 34: Marshall test*
- *Part 35: Laboratory mixing*
- *Part 36: Determination of the thickness of a bituminous pavement*
- *Part 37: Hot sand test for the adhesivity of binder on precoated chippings for HRA*
- *Part 38: Common equipment and calibration*
- *Part 39: Binder content by ignition*
- *Part 40: In situ drainability*
- *Part 41: Resistance to de-icing fluids*
- *Part 42: Amount of foreign matter in reclaimed asphalt*
- *Part 43: Resistance to fuel*
- *Part 44: Crack propagation by semi-circular bending test*
- *Part 45: Saturation Ageing Tensile Stiffness (SATS) conditioning test*
- *Part 46: Low temperature cracking and properties by uniaxial tension tests*
- *Part 47: Determination of the ash content of natural asphalts*
- *Part 48: Interlayer bonding (Torque bond test – TBT, Shear bond test – SBT, Tensile Adhesion Test - TAT)¹⁾*
- *Part 49: Determination of friction after polishing¹⁾*
- *Part 50: Resistance to scuffing¹⁾*

WARNING — The method described in this European Standard may require the use of dichloromethane (methylene chloride), 1,1,1-trichloroethane, benzene, trichloroethylene, xylene, toluene, tetrachloroethylene 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 important that adequate training be given to staff employed in the usage of these substances.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech

¹⁾ In preparation.

Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This document describes a test 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 test can be undertaken on either loose or compacted asphalt materials. The procedure is suitable for the recovery of paving grade bitumens, for which materials this European Standard is the reference method. The fractionating column procedure (see EN 12697-4) is the reference method for mixtures containing volatile matter such as cut-back bitumen.

For recovery of polymer modified bitumens, the rotary evaporator procedure is recommended.

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 12697-38, *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 terms and definitions given in EN 12697-1:2012 and the following apply.

3.1

soluble binder content

proportion 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.

Note 2 to entry: The soluble binder content is expressed in percent by mass.

3.2

insoluble binder content

proportion of binder that adheres to the aggregate after extraction

Note 1 to entry: The insoluble binder content is expressed in percent by mass.

4 Principle

The bitumen is separated from the sample by dissolving in dichloromethane (or other suitable solvent). After removal of undissolved solids from the bitumen solution, the bitumen is recovered from it by vacuum distillation using a rotary evaporator. The bitumen is in solution for less than 24 h.

5 Apparatus

5.1 Apparatus for the extraction of the soluble bitumen

A suitable container with stopper in which the sample and solvent can be agitated together, an asphalt analyser 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 approximately equivalent to the softening resulting from any solvent residue.

5.2 Apparatus for the clarification of the bitumen solution

For clarification of 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.

NOTE If an asphalt analyser is used for the extraction of soluble bitumen, the use of a centrifuge is not required.

5.2.1 Sample tube centrifuge, capable of developing an acceleration of at least 15 000 m/s² in accordance with the formula:

$$a = 1,097 \times n^2 \times r \times 10^{-5} \quad (1)$$

where

- a is the acceleration, expressed in metres per second squared (m/s²);
- n is the number of revolutions, expressed as revolution 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 this document.

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/s².

5.2.3 A pressure filter, of appropriate size.

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

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.

5.3 Distillation apparatus

NOTE The distillation apparatus, a typical rotary evaporator, is shown in Figure 1.

5.3.1 Rotary evaporator, incorporating a rotating evaporating flask which can be operated under vacuum. Some models have an inclined condenser as shown in Figure 1, but other models using vertical condensers may be used. The apparatus shall:

- a) accept a 1 l capacity evaporating flask;

- b) have a drive motor and speed control capable of rotating the evaporating flask at (75 ± 15) r/min;
- c) be capable of operating at P_2 kPa pressure where P_2 is taken from Table 1 for the solvent to be used;
- d) have an evaporation capacity of solvent at a bath temperature of $(T_1 + 5)$ °C of at least 0,85 l/h when the flask is rotated at 75 r/min where T_1 is taken from Table 1 for the solvent to be used.

5.3.2 A 1 l, pressure resistant, evaporating flask, of heat-resistant glass fitted with a ground-glass joint.

5.3.3 Oil bath for 1 l evaporating flask, capable of raising the temperature of the oil to T_3 °C where T_3 is taken from Table 1 for the solvent to be used. A high temperature silicone oil is recommended because many other oils may be irreversibly damaged above 150 °C.

5.3.4 Vacuum pump, capable of reducing the absolute pressure in a leak-proof system to P_2 kPa where P_2 is taken from Table 1 for the solvent to be used. An oil sealed vacuum pump, running in the gas ballast mode, is recommended.

5.3.5 Two pressure gauges, capable of indicating the level of reduced pressure in the distillation apparatus; one with an absolute range from 0 kPa to 100 kPa with an accuracy of $\pm 0,5$ kPa (0 mbar to 1 000 mbar ± 5 mbar) and one with an absolute range from 0 kPa to 5 kPa with an accuracy of $\pm 0,1$ kPa (0 mbar to 50 mbar $\pm 1,0$ mbar). Alternatively, a single gauge covering the required range with the specified accuracy may be used.

5.3.6 Thermometer, capable of covering the temperature range 100 °C to 200 °C with an accuracy of $\pm 0,5$ °C.

5.3.7 A suitable container for bituminous solutions.

NOTE 1 A suitable container can be a flat-bottomed glass container of 2 l or 3 l capacity.

NOTE 2 A Winchester bottle is suitable.

6 Solvent and other materials

6.1 Dichloromethane (methylene chloride) or other suitable solvent

NOTE 1 Possible solvents include toluene, tetrachloroethylene, trichlorethylene, xylene, 1,1,1-trichlorethane and benzene, although bitumen is less soluble in 1,1,1-trichlorethane than in the other solvents. The use of other alternative solvents will require determination of the equivalent distillation conditions for inclusion in Table 1.

NOTE 2 Solvents other than those listed may produce differences in the penetration, softening point, etc. of the recovered binder due to varying degrees of retained solvent softening the binder and age hardening. In particular, chlorinated solvents can have a hardening effect on bitumen.

NOTE 3 The presence of retained toluene and xylene can be detected by infra-red spectroscopy.

6.2 Petroleum jelly, glycerol or silicon grease, to seal glass joints

6.3 Silica Gel, not passing a 0,063 mm sieve.

7 Procedure

7.1 Extraction of the bitumen and removal of insoluble matter

7.1.1 Place in a suitable container an amount of the bituminous mixture that will contain between 120 g and 150 g recoverable binder. If more binder is needed to run the required binder tests, the binder recovery shall be repeated with another sample. 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 clean and all of the soluble bitumen has visibly dissolved. Alternatively, the solution of solvent and binder from the determination of the soluble binder content in accordance with EN 12967-1 or by the use of an asphalt analyser can be used.

7.1.2 Allow the bitumen solution to stand for a minimum of 10 min, and pour out the bitumen solution through a 0,063 mm sieve to remove any insoluble material. This can be achieved by either a) or b).

a) Separation by centrifuging

Remove insoluble matter from the bitumen solution by centrifuging at an acceleration of at least $15\,000\text{ m/s}^2$ 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 $25\,000\text{ m/s}^2$ 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. 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, Annex C, should be carried out occasionally on recovered bitumens to ensure that excessive mineral matter is not present.

7.1.3 During clarification of 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 but not for more than 20 h.

7.2 Assembling the apparatus

7.2.1 Assemble the apparatus as shown in Figure 1, using the minimum amount of petroleum jelly, glycerol or silicon grease to lubricate and seal the glass joints.

7.2.2 Attach clips to retain the evaporating and receiving flasks.

7.3 Distillation procedure

7.3.1 Pass cold water through the condenser.

7.3.2 Rotate the evaporating flask at (75 ± 15) r/min and lower it into the oil bath.

7.3.3 Raise the temperature of the oil bath to $(T_1 \pm 5)$ °C where T_1 is taken from Table 1 for the solvent used.

7.3.4 Reduce the pressure in the apparatus to $(P_1 \pm 5)$ kPa where P_1 is taken from Table 1 for the solvent used.

Table 1 — Distillation Conditions

Solvent		First Phase		Second Phase		Extra
Description	Boiling Point	Temperature	Pressure	Temperature	Pressure	Temperature
	°C	T_1 °C	P_1 kPa	T_2 °C	P_2 kPa	T_3 °C
Dichloromethane	40,0	85	85	150	2,0	175
1.1.1-Trichlorethane	74,1	80	30	160	2,0	185
Benzene	80,1	80	30	160	2,0	185
Trichlorethylene	87,0	90	40	160	2,0	185
Xylene	140	120	30	180	2,0	205
Toluene	110,6	110	40	160	2,0	185
Tetrachloroethylene	121	110	40	160	2,0	180

NOTE Other distillation conditions can be used for either the solvents listed above or for other suitable solvents, if the conditions recover identical binder properties (within the precision limits for the test) to that recovered with the solvents above using the listed conditions.

7.3.5 Open the induction stopcock and allow the bitumen solution to be drawn from the glass container into the evaporating flask. Adjust the rate of flow of the bitumen solution into the flask by means of the induction stopcock, so that the rate of flow into the flask is approximately equal to the rate at which distillate is flowing into the receiving flask.

7.3.6 Do not allow the volume of the bitumen solution in the evaporating flask to exceed 400 ml or the pressure to be lower than $(P_1 - 5)$ kPa where P_1 is taken from Table 1 for the solvent used.

7.3.7 If moisture is seen to be present on the surface of the bitumen solution, take care to prevent this moisture from being drawn into the evaporating flask. To achieve this, position the inlet of the induction tube as close as possible to the bottom of the container and stop the flow before any globules of water are drawn up the tube. This requirement may necessitate rejecting the last from 20 ml to 30 ml of the bitumen solution.

7.3.8 Empty the receiving flask, if necessary.

7.3.9 When all the bitumen solution has been transferred into the evaporating flask, raise the temperature of the oil bath to $(T_2 \pm 5)$ °C where T_2 is taken from Table 1 for the solvent used. Isolate the vacuum pump and allow the pressure to rise slowly to atmospheric or to the pressure to P_2 over a period of $5 \text{ min} \pm 30 \text{ s}$ where P_2 is taken from Table 1 for the solvent used.

NOTE Taking the pressure down in two steps is the reference method.

7.3.10 This procedure is best achieved by use of an auxiliary tap in the air line (see Figure 1) to ensure that air is not forcefully directed onto the hot bitumen surface.

7.3.11 Nitrogen, as an inert gas, should be inserted instead of air in order to prevent the bitumen from ageing while the pressure is being taken down.

7.3.12 Continue distillation until the evaporation of solvent is complete and the bubbling of the bitumen in the evaporating flask is finished.

NOTE 1 This is best observed by stopping the rotation of the evaporating flask momentarily.

NOTE 2 Spectrography can be used to ensure that all the solvent has been removed from the recovered bitumen.

7.3.13 Empty the distilled solvent out of the receiving flask.

7.3.14 Gradually reduce the pressure in the apparatus to $(P_2 \pm 0,2)$ kPa over a period of $3 \text{ min} \pm 30 \text{ s}$ where P_2 is taken from Table 1 for the solvent used.

7.3.15 Maintain the temperature and pressure at $(T_2 \pm 5)$ °C and $(P_2 \pm 0,5)$ kPa respectively until bubbling of the bitumen ceases.

7.3.16 If the bitumen is still bubbling after 10 min, which may be the case with some very hard bitumens, maintain the pressure of $(P_2 \pm 0,5)$ kPa and raise the temperature to $(T_3 \pm 5)$ °C where T_3 is taken from Table 1 for the solvent used. Hold this temperature until all bubbling has ceased.

7.3.17 If this higher temperature is used, bring the bath to this temperature as quickly as possible. When reporting results obtained on bitumens recovered at this higher temperature, the time required to raise the temperature to T_3 °C shall be stated together with the total time the flask was at this temperature.

7.3.18 When bubbling ceases, maintain the conditions of temperature and pressure specified in 7.3.13 or 7.3.15 for a further 10 min.

7.3.19 Stop the rotation of the flask and allow the pressure inside the apparatus to rise slowly to atmospheric.

7.3.20 This is best achieved by use of an auxiliary tap in the air line (see Figure 1) to ensure that air is not forcefully directed onto the hot bitumen surface.

7.3.21 Nitrogen, as an inert gas, should be inserted instead of air in order to prevent the bitumen from ageing while the pressure is being taken down.

7.3.22 Raise the evaporating flask clear of the bath and wipe the outside clean.

7.3.23 Remove the evaporating flask from the apparatus. Quickly wipe the flask and the inside of the flask's neck with a clean tissue and pour the contents into a suitable container. Cover the container with a loosely fitting lid.

NOTE It will often be more convenient to allow the flask to cool to a temperature appropriate to directly prepare the bitumen for test as required in Clause 8.

7.3.24 In order to avoid the possibility of significant hardening of the bitumen by the dichloromethane (or other suitable solvent), complete the total procedure (i.e. extraction and recovery) within 20 h.

7.3.25 It is advisable to keep the vacuum pump running for about 30 min after completion of the recovery to ensure the removal of any dichloromethane (or other suitable solvent) vapour from the oil.

8 Preparation of the bitumen for testing

Prepare samples of bitumen in accordance with EN 12594.

9 Test report

With reference to this document, the test report shall include the following information:

- a) name and address of the testing laboratory;
- b) unique serial number for the test report;
- c) name of the client;

- d) description and an identification of the sample, and the date of receipt;
- e) solvent used;
- f) distillation conditions T_1 °C, P_1 kPa, T_2 °C and P_2 kPa used;
- g) whether the temperature during evaporation rose above T_2 °C and the periods when it was higher (see 7.3.15);
- h) 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

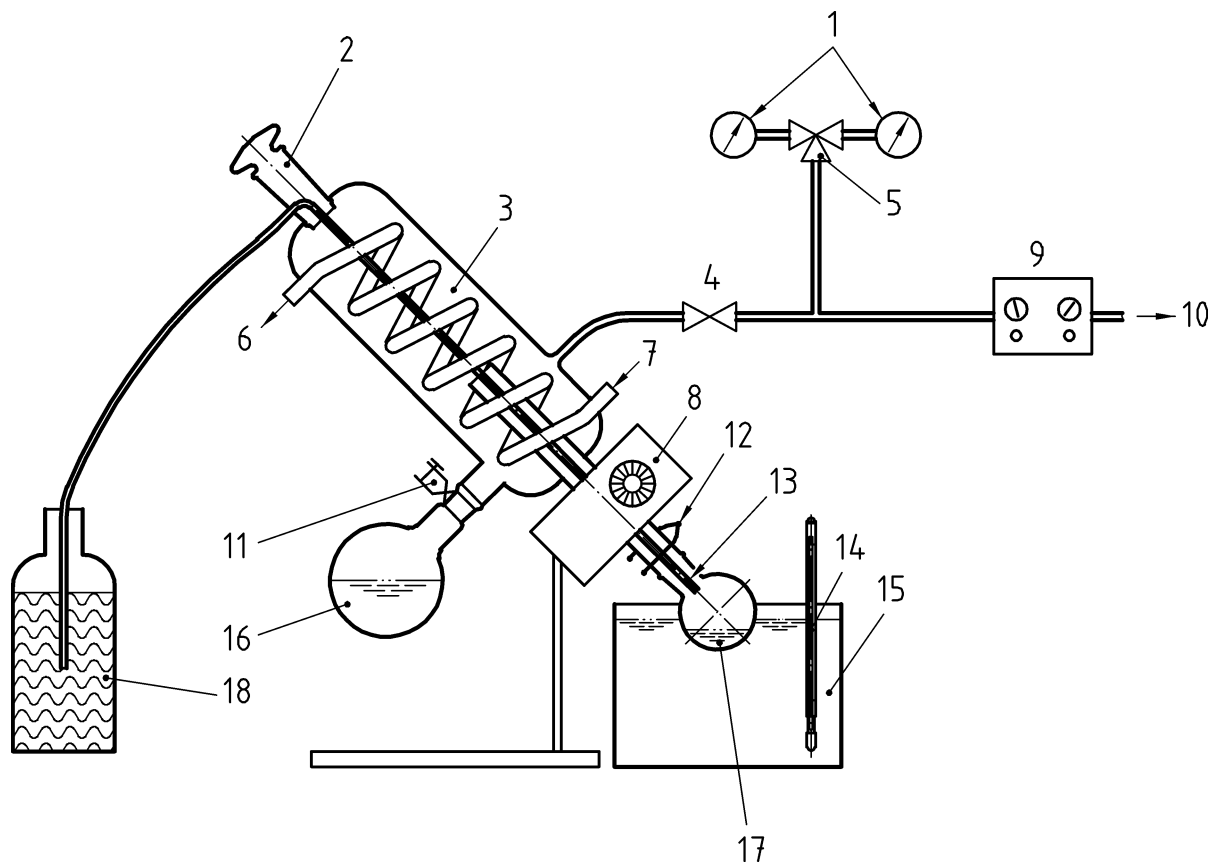
10.1 This method does not produce a result and has no precision. The precision of test methods for penetration and softening point applied to the recovered bitumen is as follows:

Table 2 — Precision values

Method		Repeatability	Reproducibility
Penetration	0,1 mm	0,10 x	0,27 x
Softening point	°C	1,9	3,4
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, as defined in EN ISO 4259, have been obtained by a statistical examination of inter-laboratory results from ten laboratories each examining four bituminous samples having original bitumen penetrations in the range from 50 to 200 penetration units.



Key

- | | | | | | |
|---|---------------------|----|--|----|----------------------------|
| 1 | vacuum gauge | 8 | rotary drive motor | 14 | thermometer |
| 2 | induction stopcock | 9 | vacuum regulator | 15 | oil bath |
| 3 | condenser | 10 | to vacuum pump | 16 | receiving flask |
| 4 | auxiliary air inlet | 11 | screw clip | 17 | rotating evaporating flask |
| 5 | change-over valve | 12 | spring clip | 18 | bitumen solution |
| 6 | water outlet | 13 | delivery tube (end approximately
1/3 of way into flask) | | |
| 7 | water inlet | | | | |

Figure 1 — A typical rotary evaporator

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*
- [3] EN ISO 4259, *Petroleum products — Determination and application of precision data in relation to methods of test (ISO 4259)*

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