BS EN 12697-1:2012

BSI Standards Publication

Bituminous mixtures — Test methods for hot mix asphalt

Part 1: Soluble binder content

National foreword

This British Standard is the UK implementation of EN 12697-1:2012. It supersedes BS EN 12697-1:2005 which is withdrawn.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 12697-1:2012) has been prepared by Technical Committee CEN/TC TC “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of an identical text or by endorsement, at the latest by December 2012, and conflicting national standards shall be withdrawn at the latest by December 2012.

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 12697-1:2005.

Compared with EN 12697-1:2005, the following changes have been made:

a) Removal of warning that the precision may be compromised with polymer-modified binders even when following Annex D;

b) For the separation of mineral, a note is added that the residue depends on the solvent and the equipment used;

c) The desiccator is made optional with the method of achieving a moisture-free atmosphere not fixed;

d) The definition of constant mass is changed;

e) Alternative procedures for determination of binder are extended;

f) Volume units are corrected from mm³ to 10³ mm³ as appropriate;

g) In Annex B, note is added to ensure the binder is well dissolved;

h) In Annex B, density of perchloroethylene is changed from (1,6 ± 0,05) g/cm³ at 24 °C to (1,6 ± 0,05) Mg/m³ at 20 ºC;

i) In Annex B, weighing of multiple sieves that are fitted to the feed funnel is allowed;

j) In Annex B, repeating the procedure with a second cup in the centrifuge is undertaken with half the flow rate;

k) In Annex B, note is added that the second run is unnecessary for some centrifuges with larger capacities;

l) In Annex C, M_2 is clarified as being the mass of fine mineral matter;

m) In Annex C, the procedures for collecting the recovered binder are clarified;

n) In Annex D, a note is added that a good solubility of the polymer-modified binder does not always guarantee a good extraction of the PmB in the bituminous mixture;

o) In Annex D, the discussion on different solvents is refined;

p) In Annex D, the flow rate is adjusted rather than just checked to avoid overflowing;

q) In Annex D, the exceptions on the apparatus and procedure for the centrifuge extractor method are removed and notes added;

r) In Annex D, note is added to continuous flow centrifuge;

s) In Annex D, precision statement is changed.

This European Standard is one of a series of standards for Bituminous mixtures as listed below:

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

EN 12697-2, *Bituminous mixtures — Test methods for hot mix asphalt — 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 for hot mix asphalt — Part 4: Bitumen recovery: Fractionating column*

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*

prEN 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 methods 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 coarse 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)]

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

prEN 12697-50, *Bituminous mixtures — Test methods for hot mix asphalt — Part 50: Scuffing resistance of surface course*[1)]

The applicability of this European Standard is described in the product standards for bituminous mixtures.

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

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1) In preparation.

Introduction

This European Standard describes a unified approach to the examination of bituminous mixtures that allows some divergence in the detail of procedures followed by individual laboratories. In Clause 5 of this European Standard, a description is given of the basic operations that together form the test method for the proper determination of the binder content of bituminous mixtures. Guidance on the test method is given in Annex A and Figure A.1, while the use of alternative items of equipment that are equally suitable for carrying out particular parts of the test method are described in Annex B. Although the apparatus specified for the separation of mineral filler from the binder solution obtained after extraction is of a suitably efficient level not to affect the precision of the test described in Clause 8, a method for determining the amount of residual mineral matter in the extract is given in Annex C for use in those particular cases where some doubt may exist.

Methods and equipment other than those described in Annex B and Annex C, including automated equipment, are permissible provided that they have been demonstrated to provide the same results as one of the methods in Annex B or Annex C within the limits of the precision given in this document. Guidance on determination of soluble binder content of mixtures with polymer-modified binders is given in Annex D.

1 Scope

This document describes test methods for the determination of the soluble binder content of samples of bituminous mixtures.

The test methods described are suitable for quality control purposes during the production of plant mix and for checking compliance with a product specification.

For the analysis of mixtures containing modified binders, the guidance of Annex D should be followed.

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

EN 12697-4, *Bituminous mixtures — Test methods for hot mix asphalt — Part 4: Bitumen recovery: Fractionating column*

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

EN 12697-28:2000, *Bituminous mixtures — Test methods for hot mix asphalt — Part 28: Preparation of samples for determining binder content, water content and grading*

EN 933-1, *Tests for geometrical properties of aggregates — Part 1: Determination of particle size distribution — Sieving method*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

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 particles after extraction

3.3
precision
closeness of agreement between independent test results obtained under stipulated conditions

Note 1 to entry: Precision depends only on the distribution of random errors and does not relate to the true value or the specified value.

Note 2 to entry: The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results. Less precision is indicated by a larger standard deviation.

Note 3 to entry: "Independent test results" means results obtained in a manner not influenced by any previous result on the same or similar test sample. Quantitative measures of precision depend critically on the stipulated conditions. Repeatability and reproducibility conditions are particular sets of extreme conditions.

3.4
repeatability
precision under repeatability conditions

3.5
repeatability conditions
conditions in which independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time

3.6
repeatability limit
maximum absolute difference between two test results obtained under repeatability conditions that may be expected with a probability of 95 %

Note 1 to entry: The symbol used for repeatability limit is r.

3.7
reproducibility
precision under reproducibility conditions

3.8
reproducibility conditions
conditions in which test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment

3.9
reproducibility limit
maximum absolute difference between two test results obtained under re…bility conditions that may be expected with a probability of 95 %

Note 1 to entry: The symbol used for reproducibility limit is R.

3.10
single test result
value obtained by applying the standard test method once, fully, on a single specimen

Note 1 to entry: The single test result may also be the mean of two or more observations or the result of a calculation from a set of observations as specified by the standard test method.

4 Preparatory treatment of laboratory samples of bituminous mixtures

Prepare laboratory samples in accordance with EN 12697-28 to obtain suitable test portions.

5 Determination of binder content

5.1 General principles of test

The test method for determining the binder content of a test portion of bituminous mixture, prepared in accordance with Clause 4, normally comprises the following basic operations:

a) binder extraction by dissolving in a hot or cold solvent;

b) separation of mineral matter from the binder solution;

c) determination of binder quantity by difference or binder recovery;

d) calculation of soluble binder content.

NOTE 1 The sequence of operations and choice of test procedures to be followed are illustrated in Figure A.1.

NOTE 2 If it is suspected that water is present in the laboratory sample, the sample should either be dried to constant mass (see Clause 6), or the water content determined by the method described in EN 12697-14, or the sample treated as in EN 12697-28.

NOTE 3 All test procedures and associated equipment relating to each basic operation shown in Figure A.1 are equally acceptable. Other equipment and procedures, including non-extraction methods, may also be used. There are documented data to show that the method and equipment will provide results with an accuracy and a precision no worse than that of one of the procedures explicitly shown in Figure A.1.

5.2 Binder extraction

5.2.1 Solvent

The tests in this European Standard require the use of solvents capable of dissolving bitumen and in some cases involve distilling the solution to recover all or some of the bitumen.

NOTE 1 Currently all hydrocarbon solvents are regarded as “hazardous” and “environmentally unfriendly” to varying degrees.

NOTE 2 Until such time as there is an agreed CEN policy with regard to the usage of hydrocarbon solvents, each member state should specify its preferred solvent, taking into account the Montreal Protocol and the views of its own Regulatory Bodies (see also "Warning" in the Foreword).

NOTE 3 Trichloroethylene should be stored in sealed bottles or canisters, which are protected against UV radiation.

NOTE 4 When trichloroethylene is recovered by distillation for further use, care should be taken to ensure that the solvent still complies with the appropriate requirements. In particular, acidity may develop; a useful precaution is to store the solvent over calcium oxide in coloured glass or suitable metal containers.

5.2.2 Apparatus

NOTE The apparatus should be calibrated and traceable.

5.2.2.1 **Balance**, capable of weighing a test portion to an accuracy of 0,05 % of its mass.

5.2.2.2 **Binder extraction apparatus**, conforming to the requirements of the method selected from B.1, as appropriate.

5.2.3 Procedure

5.2.3.1 Prepare laboratory samples in accordance with EN 12697-28 to obtain suitable test portions.

NOTE If determining binder content by difference, see Annex A.

5.2.3.2 Weigh the test portion to the nearest 0,05 % of the mass taken, and place it in the binder extraction apparatus in accordance with the requirements of the method selected from B.1, as appropriate.

5.2.3.3 The binder extraction procedure shall ensure that no soluble binder is left adhering to the aggregate particles after extraction.

NOTE In limited cases, it may be difficult to dissolve every trace of binder adhering to the aggregate (see A.4).

5.3 Separation of mineral matter

5.3.1 Apparatus

5.3.1.1 Trays, that can be heated without damage or change in mass and which are used to dry recovered aggregate.

5.3.1.2 Apparatus for the separation of mineral filler from the binder solution, conforming to the requirements of the method selected from B.2, as appropriate.

5.3.2 Procedure

5.3.2.1 Collect the binder solution obtained in accordance with 5.2 and proceed in accordance with the method selected from B.2, as appropriate.

5.3.2.2 The procedure used to separate the mineral filler from the binder solution shall ensure that the residue on ignition of the recovered binder does not exceed 0,5 %, if the nominal filler content is less than 6 % of the mass of aggregate, or 1 % if the nominal filler content is 6 % or greater, when determined in accordance with Annex C.

NOTE 1 This check is not necessary for all samples but rather serves to prove the effectiveness of the method.

NOTE 2 The residue depends on the solvent and the equipment used.

5.3.2.3 Transfer, where necessary, the clean recovered aggregate to a tray. Evaporate the solvent from the aggregate and the binder extraction apparatus. Transfer any remaining fine mineral matter from the binder extraction apparatus to the tray with the rest of the recovered aggregate, ensuring that all mineral matter has been removed from the binder extraction apparatus. Weigh and record the mass of the aggregate in the tray.

5.3.2.4 If required, determine the particle size distribution of the recovered aggregate in accordance with EN 933-1, making due allowance for any mineral filler collected by the filter paper, where appropriate.

5.4 Binder quantity

5.4.1 Apparatus

5.4.1.1 Recovery apparatus, conforming to the requirements of the method selected from B.3.

NOTE The apparatus should be calibrated and traceable.

5.4.2 Procedure

5.4.2.1 Difference method

Where the binder quantity is determined by difference, add the mass of recovered aggregate to the mass of any mineral filler collected by filter paper.

5.4.2.2 Recovery method

Where the binder quantity is determined by recovering the binder of the binder solution, follow the procedures described in B.3.

5.5 Calculation and expression of results

5.5.1 General

The soluble binder content, S, as a percentage of the mass of the original dry test portion, shall be calculated in accordance with 5.5.2, 5.5.3, 5.5.4 or 5.5.5, as appropriate.

NOTE 1 Formulae are given for un-dried test portions. Where test portions have been dried to constant mass, M, becomes the mass of the dried test portion and M_W, is deleted.

NOTE 2 For mixtures with a binder having a significant proportion of insolubles, the total binder content can be calculated by taking account of the insoluble binder content in accordance with A.4.

5.5.2 Binder determined by difference

Calculate the soluble binder content, S, in percentage by mass, by means of the following formula:

$$S = \frac{100 \times [M - (M_1 + M_W)]}{M - M_W} \quad (1)$$

where

S is the soluble binder content, expressed in percent (%);

M is the mass of un-dried test portion, expressed in grams (g);

M_1 is the mass of recovered mineral matter, expressed in grams (g);

M_W is the mass of water in the un-dried test portion, expressed in grams (g).

5.5.3 Binder by total recovery

Calculate the soluble binder content, S, in percentage by mass, by means of the following formula:

$$S = \frac{100 \times M_b}{M - M_W} \quad (2)$$

where

S is the soluble binder content, expressed in percent (%);

M is the mass of un-dried test portion, expressed in grams (g);

M_b is the mass of recovered binder, expressed in grams (g);

M_W is the mass of water in the un-dried test portion, expressed in grams (g).

5.5.4 Binder by recovery from portion (volume calculation)

Calculate the soluble binder content, S, in percentage by mass, by means of the following formula:

$$S = \frac{100 \times z \times V \times d}{(M - M_w) \times (d \times v - z)} \quad (3)$$

where

S is the soluble binder content, expressed in percent (%);

M is the mass of un-dried test portion, expressed in grams (g);

z is the average mass of binder recovered from each aliquot portion of binder solution, expressed in grams (g);

V is the total volume of solvent, expressed in cubic millimetres (mm^3);

v is the volume of each aliquot solution portion, expressed in cubic millimetres (mm^3);

d is the density of the binder at 25 °C, expressed in grams per cubic millimetres (g/mm^3);

M_W is the mass of water in the un-dried test portion, expressed in grams (g).

5.5.5 Binder by recovery from portion (mass calculation)

Calculate the soluble binder content, S, in percentage by mass, by means of the following formula:

$$S = \frac{100 \times M_B}{M - M_W} \quad (4)$$

where

S is the soluble binder content, expressed in percent (%);

M is the mass of un-dried test portion, expressed in grams (g);

M_W is the mass of the water in the un-dried test portion, expressed in grams (g);

M_B is the mass of soluble binder in the test portion, expressed in grams (g).

$$M_B = \frac{M_2 - M_1}{M_3 - M_2} M_P \quad (5)$$

where

M_P is the mass of solvent in the test portion, expressed in grams (g);

M_1, M_2, M_3 are as defined in B.3.2.

6 Drying to constant mass

6.1 General

In all the test procedures in this European Standard it is necessary, at some stage, to ensure that materials or equipment are dried to constant mass. On all such occasions, the method in 6.2 to 6.3 shall be used.

6.2 Apparatus

6.2.1 Oven or drying cabinet, of suitable capacity and capable of holding the required temperatures.

6.2.2 Balance, with an accuracy of 0,01 % or better.

6.2.3 Desiccator, of suitable capacity (optional).

6.3 Procedure

6.3.1 Place the material or equipment in the oven or drying cabinet and dry to constant mass.

NOTE 1 In case of an oven, a temperature of (110 ± 5)°C is usually suitable. Where it is necessary to dry a test portion before analysis a temperature of (80 ± 5) °C may be more suitable to avoid binder drainage, but a longer time will be necessary.

NOTE 2 In case of a drying cabinet lower temperatures are used. The lower the temperature, the longer it will take to dry to constant mass.

NOTE 3 Constant mass is defined as successive weighings after drying at least 1 h apart not differing by more than 0,1 %.

NOTE 4 For convenience, it is recommended that the successive weighings to determine constant mass should be carried out whilst the material is hot. It may be advisable to protect the balance from heat.

6.3.2 When constant mass has been achieved, cool in a moisture-free atmosphere and weigh.

NOTE A moisture-free atmosphere can be obtained by cooling in a desiccator.

7 Reporting of results

7.1 Results

Report the soluble binder content, and, where appropriate:

a) the water content to the nearest 0,1 % by mass in accordance with EN 12697-14;

b) and/or the insoluble binder content in accordance with A.4.

7.2 Test report

The report shall contain at least the following information in addition to that in 7.1:

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) identification of the test method by reference to the Annex B apparatus used;

f) any deviations, additions to or exclusions from the test method;

g) whether or not the sample was accompanied by a sampling certificate;

h) signature of officer accepting the technical responsibility for the test report;

i) date of issue.

8 Precision data

NOTE 1 Criteria for judging the acceptability of the binder content of bituminous mixtures determined by this European Standard are limited. The data that are tabulated herein were obtained from four separate precision experiments on

materials from different geographical regions within the European Union. An indication of the precision of this method of test can be obtained from the data presented in 8.1, 8.2, 8.3 and 8.4.

NOTE 2 Differences in the results obtained by two laboratories on test samples taken from the same bulk sample might be due to errors in the sample reduction and/or the procedures adopted by the analysis. Any systematic differences in the comparison of a group of test results should be investigated to detect the cause or causes of the bias.

NOTE 3 This bias will generally be due to non-adherence to the detail of the method of analysis if the same test procedures are being followed or, in isolated cases, small differences might be due to the particular test procedure selected.

NOTE 4 If differences arise when the same test procedures are being followed it will normally be possible, with co-operation between the laboratories, to eliminate the cause. If small differences occur due to the test procedures being different, it is recommended that the two laboratories concerned, at the earliest opportunity, examine the interpretation of the test results.

8.1 Precision — Experiment 1

NOTE The limits for the differences between the analysis results of two test samples obtained from the same bulk sample are as given in 8.1.4.

8.1.1 Obtain two test samples in accordance with sampling procedures in 8.1.2 to 8.1.3, as appropriate.

8.1.2 Obtain the test material for repeatability tests by dividing a sample twice the size required for a single test in accordance with the sample reduction procedure in EN 12697-28:2000.

8.1.3 Obtain the test material for reproducibility tests by first dividing a sample eight times the size required for analysis into two approximately equal portions (one for each laboratory). Each laboratory shall then reduce its portion to the size required for a single test in accordance with the sample reduction procedure in EN 12697-28:2000.

8.1.4 The repeatability, r, for binder content is 0,3 %. The reproducibility, R, for binder content is 0,5 %.

NOTE The above figures are based on a precision exercise carried out several years ago on coated macadams. The precision was found to be the same for mixtures containing aggregates of nominal sizes larger and smaller than 20 mm.

8.2 Precision — Experiment 2

The permissible range, a_{zul}, in percent, is given by:

$$a_{zul} = 2{,}77\ (0{,}10 + 0{,}002\ A) \tag{6}$$

where

a_{zul} is the permissible range, expressed in percent (%);

A is the oversize of the aggregate on a 11,2 mm test sieve, expressed in percent (%).

NOTE 1 If the individual values for two specimens do not differ by more than the permissible range, a_{zul}, the arithmetic mean should be considered to be the test result.

The repeatability standard deviation, s_r, in percent, is given by:

$$s_r = (0{,}10 + 0{,}002\ A) \tag{7}$$

where

s_r is the repeatability standard deviation, expressed in percent (%);

A is the oversize of the aggregate on a 11,2 mm test sieve, expressed in percent (%).

NOTE 2 If several values of A have been determined for a particular specimen, the arithmetic mean from these should be taken as A.

The repeatability, r, is 2,77 s_r.

The reproducibility standard deviation, s_R, in percent, is given by:

$$s_R = (0,15 + 0,002\ A) \quad (8)$$

where

s_R is the reproducible standard deviation, expressed in percent (%);

A is the oversize of the aggregate on a 11,2 mm sieve, expressed in percent (%).

NOTE 3 If several values of A have been determined for a particular specimen, the arithmetic mean from these should be taken as A.

The reproducibility, R, is 2,77 s_R.

8.3 Precision — Experiment 3

A precision experiment in accordance with ISO 5725-1 and ISO 5725-2 on the evaporation method by a fraction of the solution, a mass method, was carried out in 1994/95 with 14 participating laboratories. The precision is:

— repeatability, $r = 0,23$;

— reproducibility, $R = 0,34$ for an AC10.

8.4 Precision — Experiment 4

A precision experiment in accordance with ISO 5725-1 and ISO 5725-2 was coordinated by EAPIC in January 2008. The results of 41 participants were statistically analyzed, after which the results of 6 laboratories were rejected. All methods of EN 12697-1 were allowed and the following methods were used: hot extraction, cold extraction and continuous centrifugation (also including automated apparatus). The following precision values were obtained:

— repeatability, $r = 0,23$;

— reproducibility, $R = 0,31$;

— Average difference from real value: –0,03%.

Annex A
(informative)

Guidance on the determination of binder content

A.1 Evaluation of the results

A.1.1 The advantages and disadvantages of the various analysis methods are a matter of individual assessment which will depend on the circumstances that exist in any particular case. Nevertheless, the methods, when carried out by a skilled operator, will produce comparable test results on all materials, provided:

a) strict attention is paid to the details of the test procedures specified in this document;

b) the operator has shown that the repeatability requirements specified in Clause 8 of this European Standard have been achieved.

Comparable does not mean identical, as small systematic differences in results can arise when different techniques of analysis are used except in the case of mixtures containing volatile diluents e.g. cutback bitumens. These differences will be smaller than the reproducibility limits given in Clause 8. Where volatile diluents are present in the bituminous mixture, significant differences in analysis results could occur, depending on whether the binder is determined directly or by difference.

A.1.2 In reporting the results, the method employed should be stated. Clause 7 gives requirements for the information which is to be given.

A.1.3 If the results obtained by different laboratories are to be compared over a period of time, it is advisable to examine them for bias, which may inadvertently arise due to:

a) sampling point and method of sampling;

b) differences in method and systematic deviations from recommended technique;

c) differences in technique caused by a lack of full understanding of the test method;

d) in isolated cases, a small effect due to particular properties of the material being tested.

Where possible such differences should be reconciled before attempts are made to make direct comparison between the results of tests.

A.1.4 It should be appreciated that, compared to the bitumen in the freshly mixed material, changes can take place in the nature of bitumen in the road pavement which affect its solubility in the solvents used in these methods of analysis. This factor, together with binder loss due to trafficking, increase in aggregate content due to the ingress of uncoated detritus and changes in aggregate grading resulting from compaction or the cutting of cores and cut out samples, can affect the results of analysis of samples of old material. These points should be taken into account when considering the results of analysis of other than freshly mixed materials and particularly materials sampled after considerable trafficking.

A.2 Effect of water content

It is important in the examination of bituminous mixtures to understand fully the effect of the presence of water in the test samples. In those mixtures produced from essentially dry aggregates and sampled for immediate testing there is normally a very low water content. In materials produced from wet aggregates however, especially at the lower mixing temperatures and in materials sampled after exposure to the weather, there may be considerable quantities of water. In the methods where the binder content is determined by difference,

it is essential that an accurate estimation of the water content is available to ensure correct binder contents. Similarly, where the aggregate passing the 63 µm test sieve is determined by difference, due allowance should be made for the presence of water.

A.3 Choice of test equipment and the sequence of operations

The choice of particular items of test equipment and of the sequence of operations to obtain a test result is made in accordance with Figure A.1 by the individual laboratory management, unless otherwise specified by the purchaser. All procedures should be considered to be of equivalent status. The choice will mainly depend on cost and availability of test equipment, duration of test procedure, and purpose of test. The use of each major item of equipment permitted by this European Standard is described in Annex B.

A.4 Determination of total binder content

In the estimation of total binder content of mixtures containing insoluble binder components, it is necessary to allow for that portion of the binder which is insoluble in the solvent used. For this purpose, the total binder content, B, can be calculated by means of the following formula:

$$B = \frac{100 \times S}{100 - T} \tag{A.1}$$

where

B is the total binder content, expressed in percent by mass of the dry sample (%);

S is the soluble binder content, expressed in percent (%);

T is the proportion by mass of the binder that is insoluble in the solvent used, expressed in percent (%).

The calculation of the portion of the binder, which is insoluble, should be based on the solubility of a sample of the binder prior to mixing. The solubility should be determined in accordance with EN 12592.

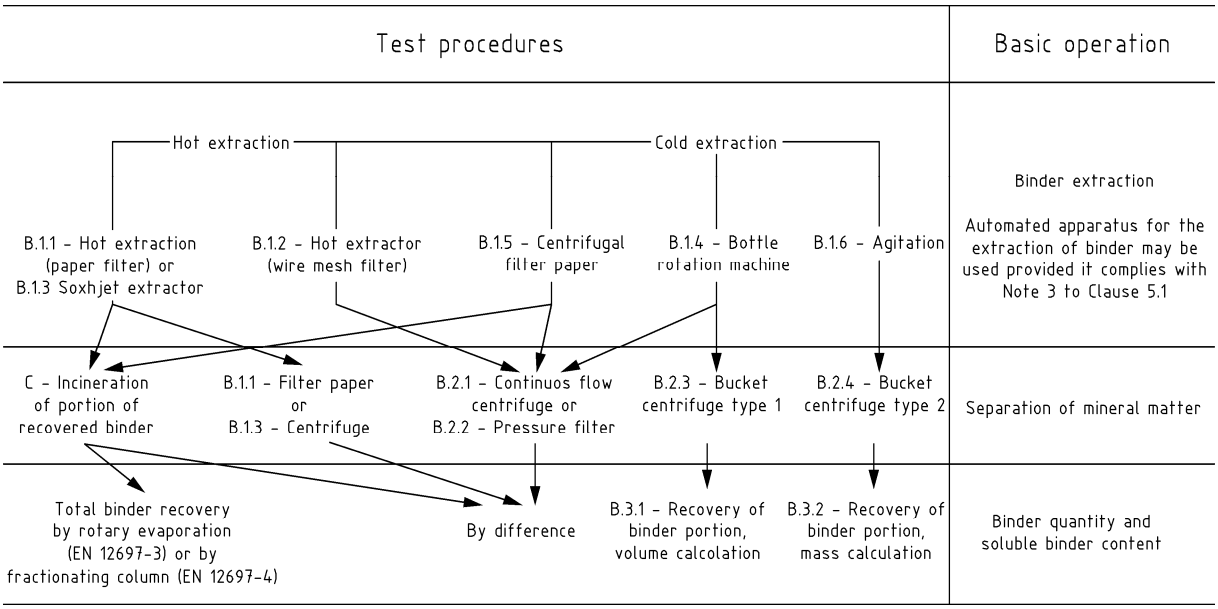


Figure A.1 — Alternative procedures for determination of binder

Annex B
(normative)

Use of equipment for the determination of binder content

B.1 Binder extraction

B.1.1 Hot extractor (paper filter) method

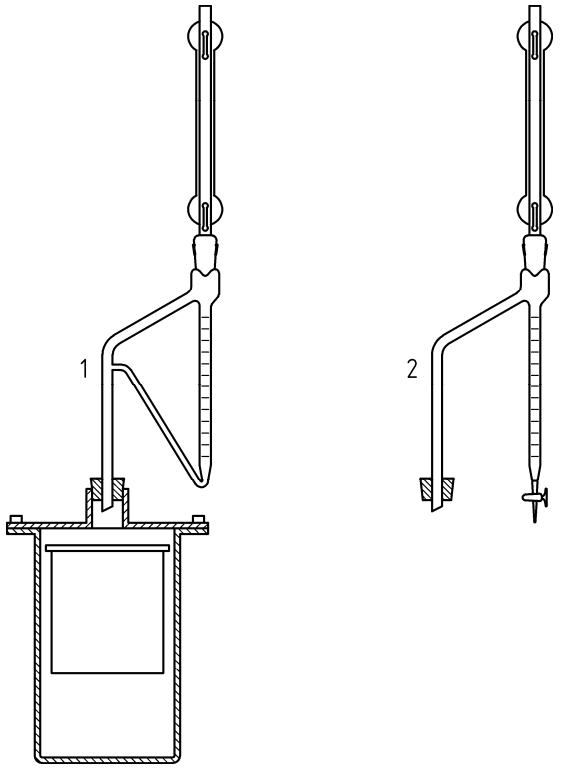
B.1.1.1 Apparatus

NOTE 1 The apparatus should be calibrated and traceable.

NOTE 2 A suitable assembly is shown in Figure B.1.

B.1.1.1.1 Hot extractor, consisting of a cylindrical container made from non-corrodible or brass gauze of about 1 mm to 2 mm aperture size or; alternatively, a spun copper tube, with a ledge at the bottom on which a removable gauze disc rests.

The container is retained in position in the top two-thirds of a metal pot. The pot is flanged and fitted with a secure cover and suitable jointing gasket. The cover is held in position so that the joint between the container and the cover is solvent tight.



Key

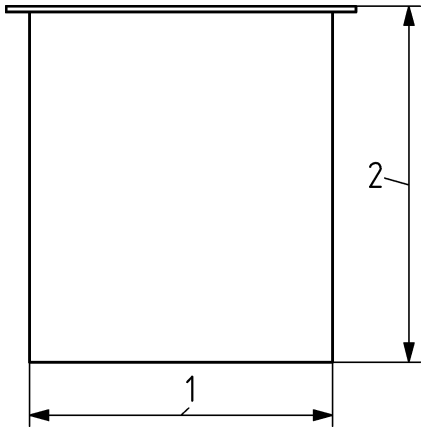
1 solvent density > 1
2 solvent density < 1

Figure B.1 — Assembled apparatus for the hot extractor method

NOTE 1 The essential features of the construction are indicated in Figures B.2 and B.3.

NOTE 2 It is advantageous to have containers and pots of more than one size, the size employed being appropriate to the quantity of material taken for analysis.

Dimensions in millimetres



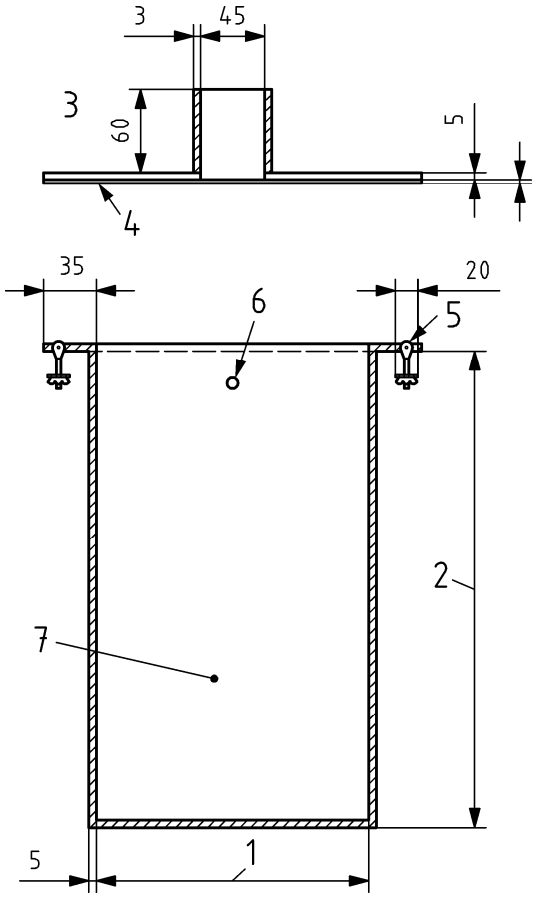
Key

1 120 mm to 200 mm as appropriate
2 120 mm to 250 mm as appropriate

Figure B.2 — Cylindrical container for the hot extractor method

B.1.1.1.2 Cylinder, of brass gauze, 1 mm to 2 mm aperture size, or of spun copper with a brass, gauze base or other non-corrodible material.

Dimensions in millimetres



Key

1 150 mm to 230 mm, as appropriate
2 200 mm to 400 mm, as appropriate
3 brass or welded steel cover
4 gasket ring
5 6 or 8 slots equally spaced around the circumference, to take swelling bolts
6 3 pegs to take gauze cylinder
7 brass or welded steel outer pot

Figure B.3 — Brazed brass or welded steel pot for the extractor method

NOTE This design has been found satisfactory but alternative designs may be employed.

B.1.1.1.3 Graduated receiver, $12{,}5 \times 10^3$ mm^3, or a receiver of similar type, suitable for use with solvents of a lower density than water, but fitted with a stopcock so that water may be drawn off into a receiver as necessary.

NOTE The receivers may be fitted with ground glass joints; in these cases an adapter may be necessary to connect the receiver to the cover of the pot.

B.1.1.1.4 Water-cooled reflux condenser, with the lower end ground at an angle of approximately 45° to the axis of the condenser.

B.1.1.1.5 Heater; such as an electric hot plate.

NOTE Gas rings should not be used because of the risk of toxic fumes arising from decomposition of any free solvent vapour and the corrosion of the pot if made of steel.

B.1.1.1.6 Filter paper.

B.1.1.1.7 **Desiccator**, of a size sufficient to accommodate the cylindrical containers fitted with filter papers.

B.1.1.1.8 **Receiver**.

B.1.1.2 Procedure

B.1.1.2.1 Fit a filter paper into the cylindrical container to form a complete lining and dry the whole at (110 ± 5) °C. Then cool in the desiccator and weigh to an accuracy of 0,1 g.

B.1.1.2.2 Carefully place the test portion in the lined container and weigh the whole to the nearest 0,05 % of the mass taken. Carry out the weighing operations involving the dried filter paper as rapidly as possible to prevent undue absorption of moisture from the atmosphere. Place the container in the pot and pour sufficient solvent (according to the size of the extractor) over the sample to permit refluxing.

B.1.1.2.3 Bolt on the cover with the dry gasket in position, and fit the receiver and condenser.

B.1.1.2.4 Heat the pot so as to avoid intense local heating, and to ensure a steady reflux rate of two to five drops per second from the end of the condenser.

NOTE Any water present in the sample will collect in the receiving tube, while the solvent will flow back over the sample and drain through the filter paper into the bottom of the pot.

B.1.1.2.5 If the amount of water collected exceeds the capacity of the receiver, discontinue the distillation, disconnect the condenser and remove a measured portion of the water by means of a pipette and pipette filler. Then re-assemble the apparatus and restart the distillation.

NOTE If a stopcock is fitted to the receiver, the water may be drawn off and measured in a receiver without interrupting the distillation process.

B.1.1.2.6 Continue heating until the extraction is complete and water ceases to collect in the receiver.

NOTE 1 The completion of extraction can only be determined reliably by dismantling the apparatus and examining the aggregates.

NOTE 2 If the apparatus is permitted to stand for more than about 2 h there is a possibility of water from the atmosphere being absorbed by the filter paper. It is therefore strongly recommended that the initial period of refluxing should be of sufficient duration to ensure that all of the water is collected without interruption except for emptying the receiver.

B.1.1.2.7 Remove the mineral aggregate with its container and dry to constant mass (see Clause 6).

NOTE For convenience, it is recommended that the successive weighings to determine constant mass should be carried out whilst the material is hot. It may be advisable to protect the balance from heat.

B.1.1.2.8 When constant mass has been achieved, cool the container and contents in a desiccator and weigh.

B.1.1.2.9 In order to remove any fine material present in the solution at the end of the test, filter the whole of the solution through a suitable grade of filter paper, or centrifuge it, and determine the mass of insoluble matter in accordance with Figure A.1.

NOTE This design has been found satisfactory but alternative designs may be employed.

B.1.2 Hot extractor (wire mesh filter) method

B.1.2.1 Apparatus

NOTE 1 The apparatus should be calibrated and traceable.

NOTE 2 A suitable assembly is shown in Figure B.4.

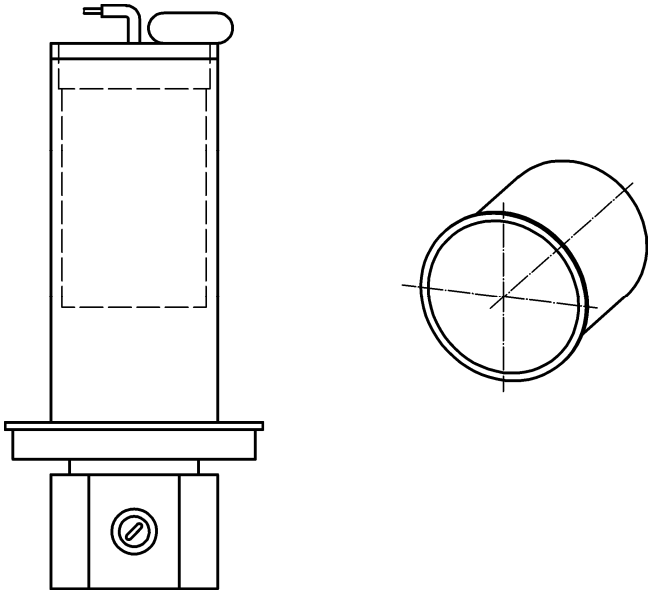


Figure B.4 — Hot extraction apparatus test set

B.1.2.1.1 **Glass and/or metal extractors**, fitted with condenser and suitable extraction cup e.g. extraction thimble wire basket made of 63 µm wire cloth or a metal cylinder with 63 µm wire cloth sieving medium, as appropriate, and a protective sieve.

B.1.2.1.2 **Flask**, either round-bottomed or flat-bottomed.

B.1.2.1.3 **Extraction thimbles**, of fibrous material.

B.1.2.1.4 **Heater**, such as an electric hot plate.

NOTE Gas rings should not be used because of the risk of toxic fumes arising from decomposition of any free solvent vapour and the corrosion of the extractor if made of steel.

B.1.2.1.5 **Desiccator**, used to cool and store the dried extraction thimbles and mineral matter.

B.1.2.2 Procedure

B.1.2.2.1 Weigh the test portion into the extraction cup and extraction thimbles made of fibrous material, which have been dried immediately prior to extraction.

B.1.2.2.2 Place the extraction cup in the extraction apparatus and extract the binder by boiling solvent until the solvent that leaves the extraction cup becomes colourless.

B.1.2.2.3 After extraction, remove the mineral aggregate with its container and dry to constant mass (see Clause 6).

B.1.2.2.4 In order to remove any fine material present in the solution at the end of the test, filter the whole of the solution through a suitable grade of filter paper, or centrifuge it, determine mass of insoluble matter in accordance with Figure A.1.

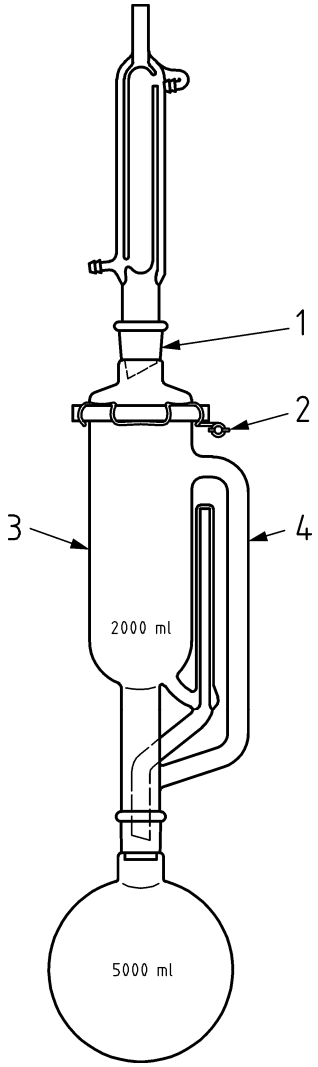
B.1.3 Soxhlet extractor method

B.1.3.1 Apparatus

NOTE The apparatus should be calibrated and traceable.

B.1.3.1.1 Glass extraction equipment, (modified Soxhlet equipment) consisting of a flask, an extractor with tap and vapour tube, and a condenser.

NOTE A suitable extractor is shown in Figure B.5.



Key

1 QUICKFIT DA 5/100 or similar
2 QUICKFIT JC 100F or similar
3 QUICKFIT EX 5/105 or similar
4 extraction case

Figure B.5 — Modified Soxhlet extractor

B.1.3.1.2 Extraction case, of pressed filter paper, or folded filter.

B.1.3.1.3 Gauze, two pieces (optional).

B.1.3.1.4 Heating equipment, such as steam bath, hot plate or heating lamps.

NOTE Gas rings should not be used because of the risk of toxic fumes arising from decomposition of any free solvent vapour.

B.1.3.1.5 **Desiccator or heated storage-box**, used to store the dried extraction case.

The heated storage-box shall be designed for the extraction cases so that, when placed close to the extractor, cases can be kept within the fume cupboard until all solvent is evaporated.

NOTE A typical storage-box is shown in Figure B.6.

Dimensions in millimetres

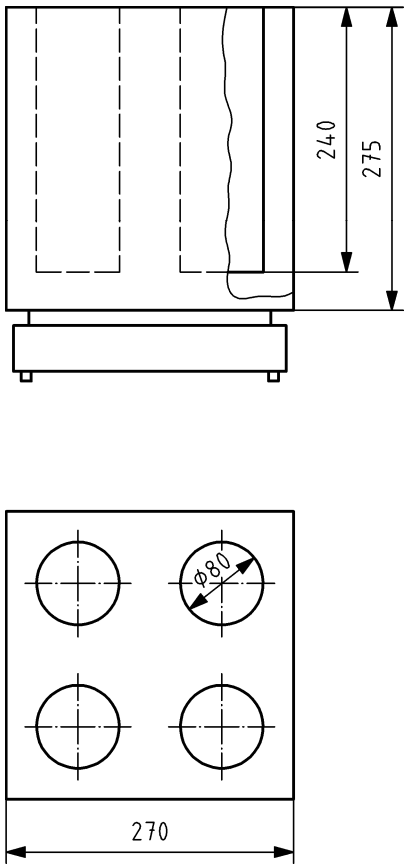


Figure B.6 — Typical heated storage-box

B.1.3.2 Procedure

B.1.3.2.1 Weigh the flask and dry extraction case, both with an accuracy of 0,05 g. Place the test portion in the extraction case and weigh the case with the test portion after it has cooled down, observing an accuracy of 0,05 g.

NOTE It is only necessary to weigh the flask when binder recovery is required.

B.1.3.2.2 Place the case with test portion on the gauze in the extractor, which has been filled with solvent to the extent that the material in the case is for the major part covered by the solvent. Place the other gauze on the case.

NOTE The amount of solvent depends on the volume of the flask.

B.1.3.2.3 Open the extractor tap.

B.1.3.2.4 Switch on the heater.

NOTE The heat supply should be such that the case can cope with the flow and does not overflow.

B.1.3.2.5 Stop the extraction when the solvent collected in the extractor becomes virtually colourless.

NOTE This may be checked by allowing the solvent in the extractor to cover the bottom part of the case. The tap is then opened and the solvent dripping from the case should be virtually colourless.

B.1.3.2.6 Remove the mineral aggregate with its container and dry to constant mass (see Clause 6).

B.1.3.2.7 In order to remove any fine material present in the solution at the end of the test, filter the whole of the solution through a suitable grade of filter paper or centrifuge it, and determine the mass of insoluble matter.

B.1.4 Bottle rotation machine method

B.1.4.1 Apparatus

NOTE The apparatus should be calibrated and traceable.

B.1.4.1.1 **Metal bottles**, of capacity appropriate to the size of sample being analysed, e.g. 600×10^3 mm^3, $2\,500 \times 10^3$ mm^3, $7\,000 \times 10^3$ mm^3, $12\,000 \times 10^3$ mm^3, with wide mouths and suitable closures.

NOTE Bottles should not be filled to more than three-quarters full.

B.1.4.1.2 **Machine**, capable of rotating the bottles about their longitudinal axes at a speed of (15 ± 5) rev/min.

B.1.4.2 Additional apparatus for procedure 2 (B.1.4.4)

B.1.4.2.1 **Volumetric flasks,** of appropriate capacity, e.g. 250×10^3 mm^3, 500×10^3 mm^3, $1\,000 \times 10^3$ mm^3 and $2\,000 \times 10^3$ mm^3.

NOTE Other systems of measuring the solvent may be employed provided that the apparatus used is calibrated.

B.1.4.2.2 **Thermometer**, capable of measuring to ±0,5 °C up to 150 °C.

B.1.4.3 Procedure 1 — Binder by difference

B.1.4.3.1 Place the test portion in a metal bottle of appropriate size.

B.1.4.3.2 Add the solvent to the test portion to give a solution of not greater than 4 % concentration of soluble binder.

NOTE 1 300×10^3 mm^3 of dichloromethane (methylene chloride or similar) should be adequate for an 800 g test portion of asphalt with 4 % to 7 % bitumen content.

NOTE 2 If dichloromethane is used, the temperature of the sample should not be above about 25 °C as a dangerously high concentration of solvent vapour may be produced. Also, pressure may build up in the bottle leading to the risk of explosion on ejection of solvent or removal of the bung.

B.1.4.3.3 Close the bottle and roll it on the bottle-rotating machine for the time specified in Table B.1.

NOTE 1 It is recommended that the rotation speed of the bottles does not exceed 20 rev/min, because of risks of crushing.

NOTE 2 For material, which has been on the road for more than one month, the extraction time might need to be extended to ensure complete extraction of the soluble binder. For freshly mixed material, the extraction time may be reduced provided that it can be shown that it yields identical results to those obtained by extraction for the minimum specified times given in Table B.1.

NOTE 3 Where degradation of the aggregate is possible due to attrition in the rolling bottle, the rolling should be intermittent but the total rolling time should not exceed 30 % of the extraction time specified in Table B.1.

NOTE 4 Verify that the binder is well dissolved. If not, the minimal extraction time in Table B.1 should be increased.

B.1.4.3.4 After the specified rolling time allow the contents to stand for at least 2 min so that filtration of the solution is made easier.

NOTE Aggregate remaining in the bottle after the solution is poured out for filtration should be shaken with a further quantity of solvent (i.e. about 0,5 l) and the process repeated until no discoloration of the solvent is visible and the washings are visibly free from material in suspension.

Table B.1 — Time required for extraction by bottle rotation machine

Type of material	**Minimum extraction time** (min)
Asphalt concrete with paving grade bitumen	20
Porous asphalt	20
Stone mastic asphalt	30
Mastic asphalt	30
Hot rolled asphalt	30
Soft asphalt	20
Coated chippings for hot rolled asphalt	10

B.1.4.4 Procedure 2 – Binder portion recovered

B.1.4.4.1 Place the test portion in a metal bottle.

B.1.4.4.2 Measure the required volume of solvent using appropriate volumetric flasks (see B.1.4.2.1) and measure and record the temperature of the solvent.

B.1.4.4.3 Add the measured volume of solvent to the sample to give a solution of between 2 % and 4 % mass concentration of soluble binder.

NOTE 1 An estimate of the total volume, V, of solvent required is given by the following formula:

$$V = \frac{M \times S_e}{C_s} \quad \text{(B.1)}$$

where

V is the estimated total volume of solvent, expressed in 10^3 cubic millimetres (mm^3);

M is the mass of the sample, expressed in grams (g);

S_e is the estimated soluble binder in the sample, expressed in percent (%);

C_s is the required mass concentration of solution, expressed in percent (%).

The estimated volume, V, should be rounded to the nearest 250×10^3 mm^3.

NOTE 2 At ambient temperatures above 25 °C, there will be an increased risk of significant inaccuracy due to evaporation.

NOTE 3 Solutions of 2 % are necessary for materials such as 20 mm coated chippings.

NOTE 4 The difference between the temperature of the solvent when measured in accordance with B.1.4.4.2 and the temperature of the centrifuged binder solution in accordance with B.3.1.2.3 should not exceed ± 3 °C.

B.1.4.4.4 Close the bottle, and roll on the bottle-rotating machine for the time specified in Table B.1.

NOTE 1 For material which has been on the road for more than one month, the extraction time might need to be extended to ensure complete extraction of the soluble binder.

NOTE 2 Where degradation of the aggregate is possible due to attrition in the rolling bottle, the rolling should be intermittent but the total rolling time should not exceed 30 % of the extraction time specified in Table B.1.

B.1.4.4.5 Remove the closed bottle from the rotating machine and stand it upright for a period of approximately 2 min to allow the bulk of the mineral matter to settle from suspension.

B.1.4.4.6 Remove the stopper carefully from the closed bottle and, without delay, transfer approximately 500×10^3 mm^3 of liquid to a clean dry pouring bottle. Transfer to the bucket centrifuge tube sufficient liquid such that after centrifuging is completed there is enough solution to provide duplicate aliquot portions.

B.1.4.4.7 Seal the remainder of the solution in the pouring bottle until aliquot portions are satisfactorily obtained.

B.1.4.4.8 At all stages of this operation, keep evaporation to a minimum by ensuring the containers have close fitting lids and solutions are poured quickly.

B.1.5 Centrifuge extractor method

B.1.5.1 Apparatus

NOTE The apparatus should be calibrated and traceable.

B.1.5.1.1 **Oven**, capable of maintaining the temperature at (110 ± 5) °C.

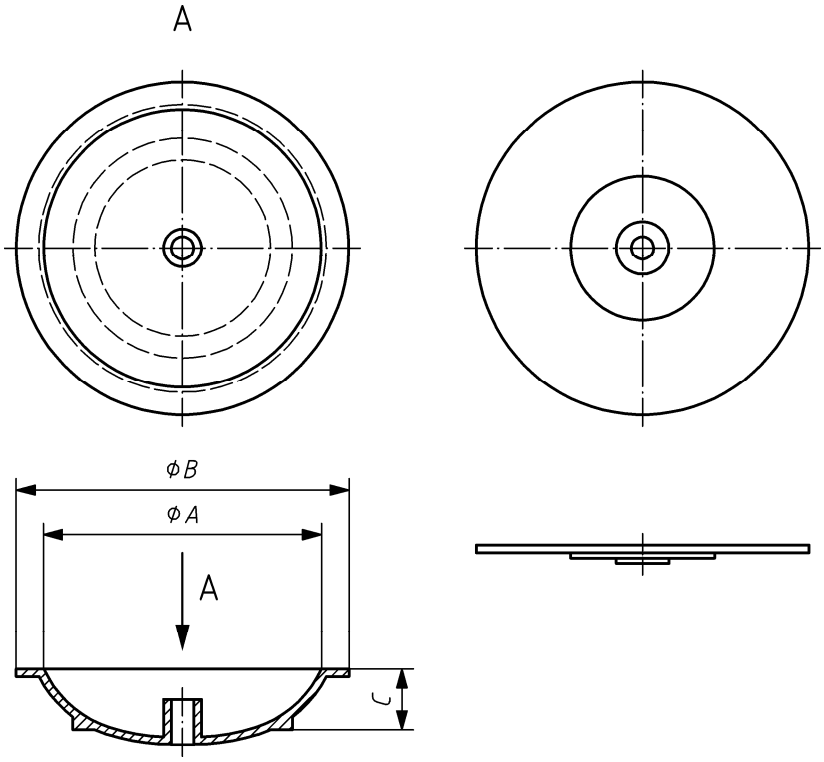
B.1.5.1.2 **Flat trays**.

B.1.5.1.3 **Volumetric flasks**, of capacity $2\,000 \times 10^3$ mm^3, $1\,000 \times 10^3$ mm^3 and 100×10^3 mm^3.

B.1.5.1.4 **Desiccator**, for cooling.

B.1.5.1.5 **Extraction apparatus**, consisting of a bowl approximating to that shown in Figure B.7 and an apparatus in which the bowl may be revolved at controlled variable speeds up to 3 600 rev/min.

The apparatus shall be provided with a container for catching the solvent thrown from the bowl and a drain for removing the solvent. The apparatus preferably shall be provided with explosion-proof features and installed in a hood or an effective surface exhaust system to provide ventilation.



Typical sample bowl dimensions (mm)			
Capacity	**A**	**B**	**C**
1 500 g	250	210	65
3 000 g	310	266	90

Figure B.7 — Typical extraction unit bowl

B.1.5.1.6 Filter rings, to fit the rim of the bowl.

NOTE Low-ash paper filter rings may be used, provided the ash content of the paper does not exceed 0,2 %.

B.1.5.2 Procedure

Follow one of the methods described in B.1.5.2.1 or B.1.5.2.2

B.1.5.2.1 Method 1

B.1.5.2.1.1 Place the weighed test portion in the bowl.

B.1.5.2.1.2 Cover the test portion in the bowl with solvent and allow sufficient time for the solvent to disintegrate the test portion, but no longer than 1 h. Place the bowl containing the test portion and solvent in the extraction apparatus. Dry the filter disk to constant mass (see Clause 6) and allow the disc to cool down in the desiccator before weighing. Determine the mass of the filter ring and fit it around the edge of the bowl. Clamp the cover on the bowl tightly and place a beaker or flask under the drain to collect the extract.

B.1.5.2.1.3 Start the centrifuge revolving slowly and gradually increase the speed to a maximum of 3 600 rev/min or until solvent ceases to flow from the drain. Allow the machine to stop, add approximately 200×10^3 mm^3 of solvent and repeat the procedure. Use sufficient 200×10^3 mm^3 additions of solvent so that the extract is virtually colourless. Collect the extract and washings in a suitable container.

NOTE The volume of the total extract should be measured if it is required to determine the residual mineral matter in the binder extract by incineration, in accordance with Annex C.

B.1.5.2.1.4 Remove the filter ring from the bowl and dry in air. If felt filter rings are used, brush off mineral matter adhering to the surface of the ring and add to the extracted aggregate. Dry the ring to constant mass in an oven at a temperature of (110 ± 5) °C.

B.1.5.2.1.5 Carefully remove all contents of the bowl, place them in a metal tray and dry them to constant mass at a temperature of (110 ± 5) °C.

NOTE The mass of the extracted aggregate should be equal to the mass of the aggregate in the tray plus the increase in mass of the filter rings.

B.1.5.2.1.6 Use the alternative procedure in B.1.5.2.1.7 instead of B.1.5.2.1.4 and B.1.5.2.1.5 when low-ash filter rings are used.

B.1.5.2.1.7 Place the aggregate and filter rings in a clean metal tray. Dry as specified in B.1.5.2.1.4. Carefully fold the dried filter ring and stand it on the aggregate. Burn the filter ring. Determine the mass of the extracted aggregate in the tray immediately after cooling to a suitable temperature.

B.1.5.2.2 Method 2

B.1.5.2.2.1 Dry the filter disk to constant mass (see Clause 6) and allow the disc to cool down in the desiccator before weighing. Fit the filter ring around the edge of the bowl, weigh and record the mass of both as M_A. Place the test portion in the bowl and the filter ring, weigh and record the total mass as M_B (in g).

NOTE The mass of un-dried test portion is calculated by means of the following formula: $M = M_B - M_A$.

B.1.5.2.2.2 Cover the test portion in the bowl with solvent and allow sufficient time for the solvent to disintegrate the test portion, but no longer than 1 h. Place the bowl containing the test portion and solvent in the extraction apparatus. Clamp the cover on the bowl tightly and place a beaker or flask under the drain to collect the extract.

B.1.5.2.2.3 Start the centrifuge revolving slowly and gradually increase the speed to a maximum of 3 600 rev/min or until solvent ceases to flow from the drain. Allow the machine to stop, add approximately 200×10^3 mm^3 of solvent and repeat the procedure. Use sufficient 200×10^3 mm^3 additions of solvent so that the extract is virtually colourless. Collect the extract and washings in a suitable container.

NOTE The volume of the total extract should be measured if it is required to determine the residual mineral matter in the binder extract by incineration, in accordance with Annex C.

B.1.5.2.2.4 Remove the filter ring from the bowl and dry in air. If felt filter rings are used, brush off mineral matter adhering to the surface of the ring and add to the extracted aggregate. Dry the ring to constant mass in an oven at a temperature of (110 ± 5) °C.

B.1.5.2.2.5 Carefully remove all contents of the bowl, place them in a metal tray and dry them to constant mass at a temperature of (110 ± 5) °C. Weigh the material and record the mass of the aggregate as M_1.

B.1.5.2.2.6 Use the alternative procedure in B.1.5.2.2.7 or B.1.5.2.2.8 instead of B.1.5.2.2.4 and B.1.5.2.2.5 when low-ash filter rings are used.

B.1.5.2.2.7 When low-ash rings filter are used, place the aggregate and filter rings in a clean metal tray and dry as specified in B.1.5.2.2.4. Carefully fold the dried filter ring and stand it on the aggregate. Burn the filter ring. Determine the mass of the extracted aggregate in the tray immediately after cooling to a suitable temperature.

B.1.5.2.2.8 After extraction, remove the bowl with all the contents (extracted aggregate and filter ring), dry it to constant mass before weighing and register the total mass as M_C (in g).

NOTE The mass of recovered mineral matter is calculated by means of the following formula $M_1 = M_C - M_A$.

B.1.6 Cold mix dissolution of bitumen by agitation

B.1.6.1 Apparatus

B.1.6.1.1 Sealed containers, of appropriate capacity (for example 2 000 $\times$ 10^3 mm^3 or 3 000 $\times$ 10^3 mm^3).

B.1.6.2 Solvent

Perchloroethylene (Tetrachloroethylene) of voluminous mass (1,6 $\pm$ 0,05) Mg/m^3 at 20 °C.

B.1.6.3 Procedure

B.1.6.3.1 Place the test portion of mass, M_r, weighed to within 1 g, in a container. The temperature of the asphalt shall be less than 90 °C.

B.1.6.3.2 Weigh a mass of solvent, M_p, corresponding to 1,6 times the mass of the sample if the latter is supposed to contain more than 5 % of binder, or 0,8 times if the latter is supposed to contain 5 % or less. For Mastic Asphalt, take between three and five times the mass of the sample.

B.1.6.3.3 Shake for at least 30 min. Stand the container up and leave to settle for at least 5 min.

B.2 Separation of mineral matter

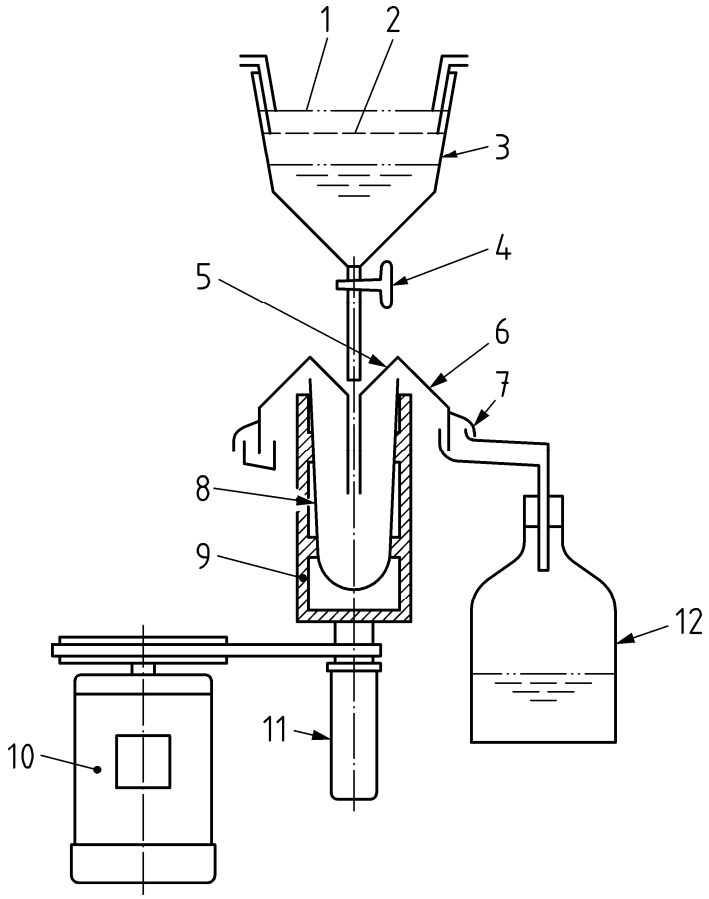
B.2.1 Continuous flow centrifuge

B.2.1.1 Apparatus

NOTE The apparatus should be calibrated and traceable.

B.2.1.1.1 High-speed continuous flow centrifuge, capable of achieving a sufficiently high rotation speed to result in a centrifugal acceleration of the order of 3 $\times$ 10^4 m/s^2 to the inner wall of the cup (see Figure B.8).

B.2.1.1.2 Funnel, fitted with a 63 µm wire cloth-sieving medium.



Key

1 2 mm sieve (optional)
2 0,63 µm sieve
3 feed funnel
4 flow-regulating tap
5 funnel introducing the liquid into the centrifuge cup
6 cap catching the solution and covering it to 7
7 centrifugate collector
8 removable aluminium cup
9 rotating cup holder
10 electric motor
11 support pin
12 liquor receiver

Figure B.8 — Schematic diagram of a continuous flow centrifuge separating the filler

B.2.1.2 Procedure

B.2.1.2.1 Separately weigh two clean and dry centrifuge cups to the nearest 0,1 g. Place one of the cups in the centrifuge and retain the other. Weigh the sieve(s) that is (are) fitted to the feed funnel.

B.2.1.2.2 Fit the feed funnel centrally above the centrifuge funnel and carefully pour the binder solution obtained from the binder extraction process into the feed funnel.

B.2.1.2.3 Open the feed funnel tap and adjust it to give a flow rate of the order of 100×10^{-6} m^3/min into the running continuous centrifuge.

B.2.1.2.4 Re-wash the filler collected in the centrifuge cup using as small a quantity of solvent as possible until the decanted solvent becomes virtually colourless.

B.2.1.2.5 Collect the centrifuged effluent. Remove the cup containing the extracted filler and place it in the oven for drying at (110 ± 5) °C.

B.2.1.2.6 Place the second cup in the centrifuge and repeat the procedure described in B.2.1.2.2 to B.2.1.2.4, except that the feed funnel tap shall be adjusted to give a flow rate of approximately half of the flow rate used in B.2.1.2.3 into the running centrifuge. After the centrifuging is completed, remove the cup and feed funnel sieve(s) and place them with the first cup in the oven for drying (optional).

NOTE The second run is unnecessary for some centrifuges with larger capacities.

B.2.1.2.7 After drying to constant mass (see Clause 6), re-weigh the two cups to calculate the filler collected by the difference in masses. Re-weigh the sieve(s) to determine the amount of mineral matter retained.

B.2.1.2.8 If required, add all the mineral matter deposited during centrifuging to the aggregate recovered by the binder extraction process.

B.2.2 Pressure filter

B.2.2.1 Apparatus

NOTE The apparatus should be calibrated and traceable.

B.2.2.1.1 **Pressure filter of appropriate size and an air pump**, for supplying dry oil-free air at a pressure of at least 2 bar.

If a filter aid is employed it shall be used in accordance with B.2.2.3.

NOTE A 3 l capacity pressure filter taking a filter paper of 270 mm diameter is suitable.

B.2.2.1.2 **Filter papers**, to fit the pressure filter.

B.2.2.1.3 **Funnel**, capable of supporting three 200 mm or 300 mm diameter sieves over the filling orifice of the pressure filter.

B.2.2.1.4 **Test sieves**, appropriately 200 mm or 300 mm diameter, complying with the requirements of ISO 3310-1 or ISO 3310-2.

B.2.2.2 Procedure

B.2.2.2.1 Weigh a dry filter paper to the nearest 0,1 g and fit it into the pressure filter. If required, support a nest of appropriate test sieves above the funnel mounted above the pressure filter. The finest test sieve shall be 63 µm size.

B.2.2.2.2 Decant the binder solution obtained from the binder extraction process through the test sieves into the pressure filter. Force the solution through the filter paper using air pressure.

B.2.2.2.3 Continue to rinse the receptacle containing the washed aggregate, in stages, to remove as much of the mineral matter as possible and pass the washings through the sieves and the pressure filter until the solvent is clean.

NOTE An alternative procedure is to carefully transfer the recovered aggregate in stages, rinsing each time, on the nest of sieves where washing is continued until the solvent is clean.

B.2.2.2.4 Transfer the clean aggregate from sieves to a tray. Evaporate the solvent from the aggregate, the sieves and the receptacle. Transfer any mineral matter in the receptacle to the tray with the remainder of the aggregate, taking care that all aggregate is removed from the receptacle and sieves.

NOTE It will usually be necessary to brush the dried sieves to ensure full aggregate recovery.

B.2.2.2.5 Weigh and record the mass of the aggregate in the tray.

B.2.2.2.6 Remove the filter paper, complete with the mineral matter that passed the 63 µm test sieve, from the pressure filter.

B.2.2.2.7 Dry to constant mass (see Clause 6); this will include the insoluble portion of the binder if insolubles were present in the binder. Deduct the mass of the filter paper.

B.2.2.3 Use of a filtering aid

NOTE In this method, the use of a filter aid, dried to constant mass, consisting of a purified diatomaceous earth is permissible.

Set the pressure filter up with a filter paper in the usual way but immediately after the specified shaking time and prior to filtration, pre-coat the filter paper as follows.

a) Weigh $(45 \pm 0{,}1)$ g of filter aid into a $1\,000 \times 10^3$ mm^3 beaker, add about 700×10^3 mm^3 of solvent and stir well.

b) In some instances, greater or lesser quantities of filter aid may be necessary. If such quantities are used they should be accurately recorded.

c) Immediately after pour the filter aid/solvent mixture into the pressure filter. Wash the beaker clean of any remaining filter aid with a few millilitres of solvent and pour into the pressure filter.

d) Apply about 0,1 bar pressure to the pressure filter until the bulk of the solvent has passed through, then release the pressure. Do not allow the pre-coat of filter aid so formed to dry.

e) Continue the procedure as described in B.2.2.2.2 to B.2.2.2.6 but when calculating the result, allow for the total mass of filter used.

B.2.3 Bucket type centrifuge — Type 1

B.2.3.1 Apparatus

NOTE The apparatus should be calibrated and traceable.

B.2.3.1.1 **Centrifuge**, typical and suitable with four buckets fitted and with centrifuge tubes of at least 50×10^3 mm^3 capacity and capable of an acceleration of between $1{,}5 \times 10^4$ m/s^2 and $3{,}0 \times 10^4$ m/s^2.

The centrifuge shall be used and maintained. The relevant recommended reduced maximum speed of operation of the centrifuge related to solution density shall be obtained from the manufacturer and shall not be exceeded when centrifuging solutions with a relative density greater than 1,0. This lower speed shall be used when calculating n in the formula given below.

The tubes shall be closed with caps such that no loss of solvent occurs during centrifuging.

NOTE 1 The tubes and caps should be checked regularly for signs of wear and replaced as necessary.

The time of centrifuging shall be obtained from Figure B.9 after calculating the acceleration, *a*, developed in the machine in question in accordance with the following formula:

$$a = 1{,}097 \times n^2 \times r \times 10^{-5} \qquad \text{(B.2)}$$

where

a is the calculated acceleration in the machine, expressed in metres per squared second (m/s^2);

n is the angular velocity, expressed in revolutions per minute (r/min);

r is the radius to the bottom of the tubes (internal), when rotating, expressed in millimetres (mm).

NOTE 2 A cooled centrifuge may be necessary to meet temperature restraints.

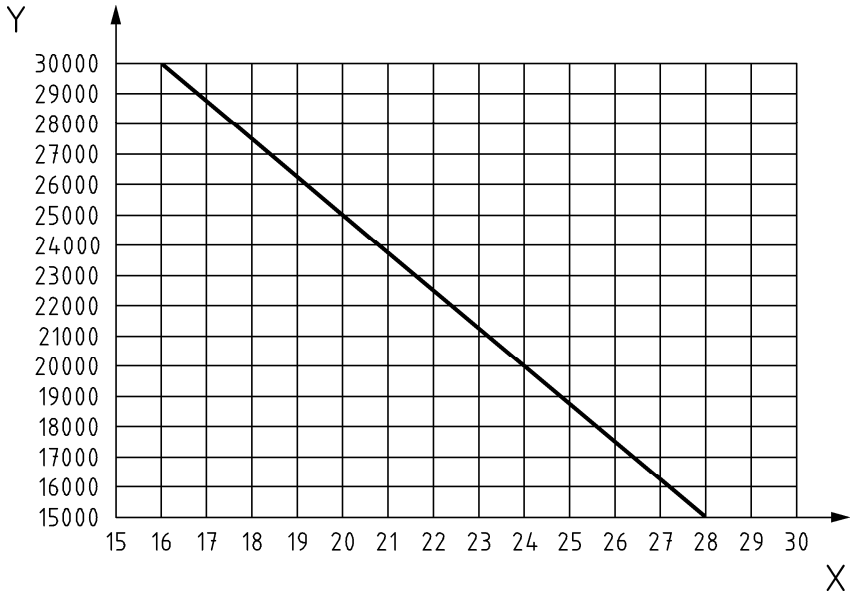
B.2.3.1.2 Pouring bottles, with a capacity between 600×10^3 mm^3 to $1\,000 \times 10^3$ mm^3 made of polypropylene and with a screw cap.

B.2.3.2 Procedure

B.2.3.2.1 Without delay, transfer approximately 500×10^3 mm^3 of binder solution obtained from the binder extraction process to a clean dry pouring bottle.

B.2.3.2.2 Transfer to the centrifuge tube sufficient liquid such that after centrifuging is complete there is enough solution to provide duplicate aliquot portions. Seal the remainder of the solution in the pouring bottle until aliquot portions are satisfactorily obtained.

B.2.3.2.3 Seal the centrifuge tube and centrifuge for the appropriate time determined from Figure B.9. At all stages of this operation, keep the evaporation to a minimum by ensuring the containers have close fitting lids and solutions are poured quickly.



Key

Y acceleration, m/s^2
X centrifuging time, min

Figure B.9 — Acceleration/time relationship for bucket centrifuge

B.2.4 Bucket type centrifuge — Type 2

B.2.4.1 Apparatus

NOTE The apparatus should be calibrated and traceable.

B.2.4.1.1 Bucket centrifuge, capable of producing a minimum acceleration of 40 000 m/s^2 fitted with buckets having a capacity greater than 20 000 mm^3.

B.2.4.2 Procedure

Centrifuge at not less than 40 000 m/s^2 for at least 30 min for Mastic Asphalt and for at least 15 min for other types of mixture.

B.3 Soluble binder content

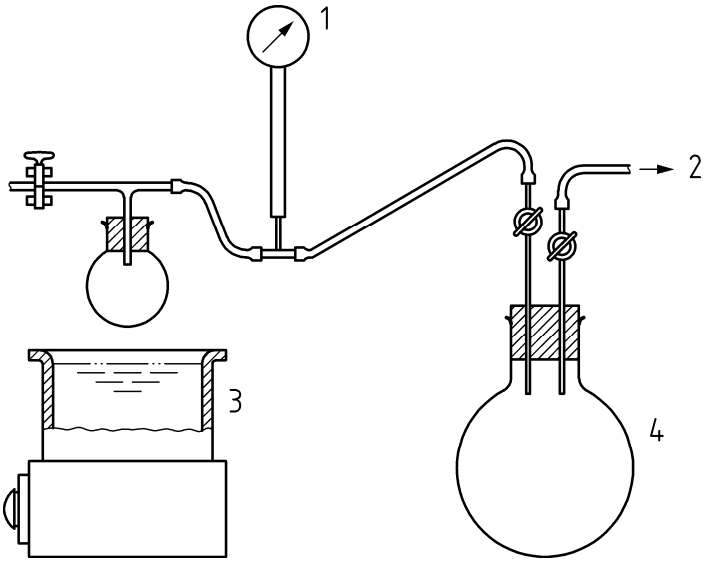
B.3.1 Method by recovery from a portion using a volume calculation

B.3.1.1 Apparatus

NOTE The apparatus should be calibrated and traceable.

B.3.1.1.1 Recovery apparatus, comprising a water bath with an electric heater capable of maintaining boiling water in the bath throughout the recovery procedure, flasks of 200×10^3 mm^3 or 250×10^3 mm^3 capacity, a pressure gauge, a vacuum reservoir and a method of maintaining reduced pressure, e.g. a filter pump.

NOTE Figure B.10 illustrates a typical arrangement for a recovery apparatus. Nevertheless, this particular arrangement is not the only one that can be used, provided that the conditions for recovering the binder from the solution as specified in B.3.1.2 are strictly followed.



Key

1 pressure gauge
2 to vacuum pump
3 water bath
4 vacuum reservoir

Figure B.10 — Recovery apparatus showing necessary features

B.3.1.1.2 Burette, of suitable size.

B.3.1.1.3 Balance, readable to 1 mg for weighing flasks.

B.3.1.1.4 Desiccator, to store the extraction flasks before weighing.

B.3.1.2 Procedure

B.3.1.2.1 Carry out the procedure in duplicate.

NOTE When the binder content of the sample cannot be estimated, it is advisable to recover the binder from one aliquot portion of solution before proceeding with the duplicate recovery to ensure compliance with the restrictions on mass contained in B.3.1.2.3.

B.3.1.2.2 Dry a flask and weigh the dry flask to the nearest 1 mg.

B.3.1.2.3 Measure and record the temperature of the solution in the centrifuge tube and then, provided the temperature is not greater than 3 °C from the temperature in B.1.4.4.2, immediately poor into the burette. Alternatively, cap the tube and allow the solution to equalise to within 3 °C, then pour into the burette. Measure a sufficient amount of the centrifuged solution into the flask, to give a residue of 750 mg to 1 250 mg of soluble binder after evaporation of the solvent.

NOTE 1 The difference between the temperature of the solvent when measured in accordance with B.1.4.4.2 and the temperature of the centrifuged binder solution in accordance with this sub-clause should not exceed ± 3 °C.

NOTE 2 An estimate of the volume, v, in cubic millimetres (mm^3), of solution (aliquot portion) required is given by the following formula:

$$v = \frac{100 \times V}{M \times S_E} \quad \text{(B.3)}$$

where

v is the estimated volume of solution (aliquot portion), expressed in cubic millimetres (mm^3);

V is the total volume of solvent, expressed in cubic millimetres (mm^3);

M is the mass of the sample, expressed in grams (g);

S_E is the estimated soluble binder in the sample, expressed in mass percent (%).

The estimate of the volume, v, should be rounded to the nearest 5 000 mm^3.

B.3.1.2.4 Remove the solvent from the binder solution by connecting the flask to the recovery apparatus (see B.3.1.1.1), immersing the flask to approximately half its depth in the boiling water, and distilling off the solvent. While the distillation is proceeding, gently shake the flask in a rotary motion so that the binder is deposited in a thin layer on the walls of the flask. Do not allow pressure above atmosphere to develop in the flask during the evaporation of the solvent.

NOTE 1 It is recommended that the distillation be carried out under reduced pressure. If reduced pressure is used, this pressure should be not less than 60 kPa.

NOTE 2 Two flasks may be connected to the vacuum system and the distillation may be carried out simultaneously provided that both are rotated continuously. Rotary evaporators may be used to agitate the flasks; they also alleviate frothing.

B.3.1.2.5 As there is relatively rapid reduction in pressure when the bulk of the solvent has been removed and frothing usually occurs at this stage, proceed as follows:

a) For penetration grade bitumen, reduce the pressure to between 18 kPa and 22 kPa in 1 min to 2 min and maintain at this pressure for a further 3 min to 4 min. The combined time under reduced pressure shall be (300 ± 15) s.

b) For cutback bitumens, allow the pressure to increase to approximately atmospheric pressure and then reduce n to between 55 kPa and 65 kPa in 1 min to 2 min and maintain it at this pressure for a further 3 mm to 4 min. The combined time under reduced pressure shall be (300 ± 15) s.

B.3.1.2.6 Remove the flask from the bath and admit air to the apparatus to increase the pressure to atmospheric. Wipe the flask dry and disconnect it, taking care to prevent the entry into the flask of water that

may have collected at the joint between the flask and the stopper. Remove all traces of solvent that remain in the flask by a gentle current of clean, oil-free and water-free air at ambient temperature. Insert the air supply tube into the flask to below mid-depth. Thoroughly clean the outside of the flask and also remove any rubber adhering to the inside of the flask neck if rubber bungs are used.

B.3.1.2.7 Cool the flask in a desiccator (see B.3.1.1.4) and weigh to the nearest 1 mg.

B.3.1.2.8 If the mass of soluble binder recovered is not between 750 mg and 1 250 mg (inclusive), repeat the procedure from B.3.1.2.1 to B.3.1.2.7 inclusive with another portion of the solution after adjusting the volume of the aliquot (see B.3.1.2.3) to ensure that an adequate mass of binder is recovered.

B.3.1.2.9 If the difference between the duplicate recoveries is greater than 20 mg, reject these results and repeat the recovery of the binder from further aliquot portions in duplicate. Use the average of these duplicate determinations to calculate the binder content.

B.3.2 Method by recovery from a portion using a mass calculation

B.3.2.1 Apparatus

NOTE The apparatus should be calibrated and traceable.

B.3.2.1.1 **Flat trays**, fitted with a cover and of minimum internal diameter 90 mm and maximum external diameter 105 mm and with an edge of (25 ± 5) mm and of mass M_1, known to the nearest 10 mg.

B.3.2.1.2 **Evaporator**, consisting of a hot plate capable of maintaining the sample at a temperature measured between 140 °C and 150 °C and an evaporation bell enabling a thermometer, linked to an aspiration device of at least 10 kPa.

Alternatively, an oven capable of maintaining the sample between 140 °C and 150 °C and fitted with an air socket able to renew the supply from the oven 3 times every 15 min.

B.3.2.1.3 **Desiccator**.

B.3.2.2 Procedure

B.3.2.2.1 After centrifuging, take a sample of $(10\,000 \pm 500)$ mm^3 with a pipette and pour onto the tray. Cover immediately. Weigh the tray with the cover as M_3 to the nearest 10 mg.

B.3.2.2.2 Either:

— Method using an evaporator: Place the tray on the hot plate so that it is horizontal. Remove the cover. Place the bell on the tray. Measure the temperature (5 ± 2) mm above the liquid in the tray. Reduce the pressure by not less than 10 kPa. When the temperature has reached a maximum, then a minimum and then the previous maximum +5 °C, remove the tray.

— Method using an oven: Preheat the oven to (150 ± 10) °C for at least 15 min, where the temperature is measured inside the oven at (5 ± 2) mm above the liquid in the tray. Place the tray horizontally in the oven, evaporate for (25 ± 2) min and remove the tray.

B.3.2.2.3 Keep the tray in a desiccator until cool. Weigh the cooled tray with its cover as M_2 to the nearest 10 mg.

B.3.2.2.4 Repeat the operations in B.3.2.2.1 to B.3.2.2.3 at least once.

Annex C
(normative)

Determination of residual mineral matter in the binder extract by incineration

C.1 General

Determine the amount of residual mineral matter in the binder extract by one of the methods in C.2 or C.3.

C.2 Method 1

NOTE The apparatus should be calibrated and traceable.

C.2.1 Apparatus

C.2.1.1 **Balance**, capable of measuring to an accuracy of 1 mg.

C.2.1.2 **Steam bath or electric hot plate**, with adjustable heating rate.

C.2.1.3 **Graduated cylinders**, of $2\,000 \times 10^3$ mm^3, $1\,000 \times 10^3$ mm^3 and 100×10^3 mm^3 capacity.

C.2.1.4 **Furnace**, capable of heating to 600 °C.

C.2.1.5 **Ignition dish**, of 125×10^3 mm^3 capacity.

C.2.1.6 **Desiccator**, of suitable capacity.

C.2.2 Reagent

Saturated solution of reagent grade ammonium carbonate $(NH_4)_2\,CO_3$.

C.2.3 Procedure

C.2.3.1 Determine the volume of the total binder solution collected in accordance with 5.2 and 5.3.

C.2.3.2 Condition the ignition dish in the furnace, cool in a desiccator and determine the mass of ignition dish to 1 mg.

C.2.3.3 Agitate the extract thoroughly and immediately measure approximately 100×10^3 mm^3 into the ignition dish.

C.2.3.4 Evaporate to dryness on a steam bath or hot plate.

C.2.3.5 Place the sample in the furnace and ash residue at a dull red heat (500 °C to 600 °C), cool, weigh and add 5×10^3 mm^3 of saturated ammonium carbonate solution per gram of ash.

C.2.3.6 Digest at room temperature for 1 h. Dry to constant mass (see Clause 6), cool in a desiccator, and determine the mass to 1 mg.

C.2.3.7 Calculate the mass of fine mineral matter in the total volume of extract, M_2, in grams, by means of the following formula:

$$M_2 = G \times \frac{V_1}{V_1 - V_2} \quad \text{(C.1)}$$

where

G is the mass of ash in aliquot, expressed in grams (g);

V_1 is the total volume of extract, expressed in cubic millimetres (mm^3);

V_2 is the volume of extract after removing aliquot, expressed in cubic millimetres (mm^3).

C.2.3.8 Calculate the soluble binder content, S, by means of the following formula:

$$S = \frac{[(M - M_W) - (M_1 + M_2)] \times 100}{M - M_W} \quad \text{(C.2)}$$

where

S is the soluble binder content, expressed in percent (%);

M is the mass of un-dried test portion, expressed in grams (g);

M_W is the mass of water in the un-dried test portion, expressed in grams (g);

M_1 is the mass of recovered mineral matter, expressed in grams (g);

M_2 is the mass of fine mineral matter in the total volume of the extract, expressed in grams (g).

C.3 Method 2

C.3.1 Apparatus

NOTE The apparatus should be calibrated and traceable.

C.3.1.1 **Balance**, capable of measuring to an accuracy of 10 mg.

C.3.1.2 **Steam bath, hot plate or heating lamps**, with adjustable heating rate.

C.3.1.3 **Ignition dish**, of 125×10^3 mm^3 capacity.

C.3.1.4 **Furnace**, capable of heating to 600 °C.

C.3.1.5 **Desiccator**, of suitable capacity.

C.3.2 Procedure

C.3.2.1 Collect the recovered binder in accordance with B.3.2 of this standard or with either EN 12697-3 or EN 12697-4. Weigh and transfer approximately one tenth to the pre-weighed ignition dish and weigh to an accuracy of 10 mg.

C.3.2.2 Incinerate the binder and heat gradually to cause the binder to carbonise, making sure it does not burn.

C.3.2.3 Heat up to a dull red heat (500 °C to 600 °C) until constant mass.

C.3.2.4 Cool the mass in a desiccator and weigh the ash to an accuracy of 10 mg.

C.3.2.5 Calculate the soluble binder content, S, in percent, by means of the following formula:

$$S = \frac{\left(M_{\mathrm{b}} - \left(\frac{M_{\mathrm{b}} \times G}{c}\right)\right) \times 100}{M - M_{\mathrm{W}}} \qquad \text{(C.3)}$$

where

S is the soluble binder content, expressed in percent (%);

M is the mass of the un-dried test portion, expressed in grams (g);

M_{W} is the mass of water in the un-dried test portion, expressed in grams (g);

M_{b} is the mass of recovered binder, expressed in grams (g);

c is the mass of incinerated binder, expressed in grams (g);

G is the mass of ash, expressed in grams (g).

C.3.2.6 Calculate the mass of fine mineral matter in the total volume of extract, M_2, in grams in accordance with C.2.3.7.

Annex D
(informative)

Guidance on determination of soluble binder content of mixtures with polymer-modified binders

D.1 General

This annex gives guidelines for the determination of the soluble binder content of samples of bituminous mixtures containing polymer-modified binders.

NOTE The producer of the polymer-modified binder should be consulted regarding the most appropriate solvent to be used.

D.2 Preparatory treatment of laboratory samples of bituminous mixtures

The same preparatory treatments of samples apply as described in Clause 4.

D.3 Determination of binder content

D.3.1 General principles of test

The same general principles as described in 5.1 apply.

D.3.2 Binder extraction

D.3.2.1 Solvent

Not all solvents used to dissolve bitumen are suitable for dissolving any polymer-modified binder. The use of an appropriate solvent suitable to dissolve the polymer binder completely is therefore very important. Insufficient solubility leads to incorrect results and to large discrepancies between the results of different methods. Therefore, a preliminary check of the solubility of the polymer-modified binder in the applied solvent is required, according to EN 12592.

NOTE 1 A good solubility of the polymer-modified binder according to EN 12592 does not always guarantee a good extraction of the PmB in the bituminous mix. Toluene, trichloroethylene, dichloromethane (methylene chloride) and perchloroethylene (tetrachloroethylene) are generally able to dissolve styrene butadiene styrene (SBS) modified binders. Toluene, trichloroethylene and perchloroethylene (tetrachloroethylene) are sometimes suitable for ethylene vinyl acetate (EVA) modified binders. Dichloromethane is generally not suitable to dissolve EVA. Few solvents have been found suitable to dissolve atactic polypropylene (APP) modified binders completely; the best solubility is generally found with hot xylene. Hot 1,3,5-trichlorinebenzene is also suitable for APP, but its use is limited to the difference method, because of the high boiling point.

NOTE 2 Warning on the use of these solvents is given in the Foreword.

NOTE 3 The solubility of polymer-modified binders may depend largely on the temperature of the solvent. This can lead to considerable differences between the results of cold and hot extraction methods.

NOTE 4 Table D.1 gives the boiling point of different solvents.

Table D.1 — Boiling points of different solvents

Solvent	Boiling point (°C)
Toluene	110,6
Trichloroethylene	87,0
Xylene	140
1,1,1-Trichloroethane	74,1
Dichloromethane	39,5
Benzene	80,1
Perchloroethylene (Tetrachloroethylene)	121

NOTE 5 The temperature dependent solubility may lead to a precipitation of the polymer on the recipient after cooling.

NOTE 6 Hot solvents are not suitable for all procedures described hereafter.

D.3.2.2 Apparatus and procedure

D.3.2.2.1 General

The apparatus and procedure used for the binder extraction are given in 5.2.2 and 5.2.3 respectively. Special conditions related to the different methods are recommended for mixtures containing polymer-modified binders. They are described in D.3.2.2.2 to D.3.2.2.6.

D.3.2.2.2 Hot extractor (paper filter and wire mesh filter) method

The apparatus and procedure for the hot extractor method are given in B.1.1 (paper filter) and B.1.2 (wire mesh filter), except that extraction should not be stopped when the solvent collected in the extractor becomes virtually colourless. Extraction should be continued for a period of approximately 10 % of the time necessary to obtain a colourless collected solvent.

It is recommended that the apparatus includes a transparent inspection window in order to determine the completion of extraction.

D.3.2.2.3 Soxhlet extractor method

The apparatus and procedure for the soxhlet extractor method are given in B.1.3, except that extraction should not be stopped when the solvent collected in the extractor becomes virtually colourless. Extraction should be continued for a period of approximately 10 % of the time necessary to obtain a colourless collected solvent.

Adjust the flow rate so as the solvent does not overflow.

NOTE Overflow of the case may more easily occur with polymer-modifier binder, because of clogging of the polymer in the filter.

D.3.2.2.4 Bottle rotation machine method

The apparatus and procedure for the bottle rotation machine method are given in B.1.4, except that the minimum rolling time given in Table D.2 should be respected. The minimum extraction time may be adjusted in accordance with the producer of the polymer-modified binder.

Table D.2 — Time required for extraction by bottle rotation machine

Type of material	Minimum extraction time (min)
Asphalt concrete	30
Porous asphalt	30
Stone mastic asphalt	40
Mastic asphalt	40
Hot rolled asphalt	40
Soft asphalt	30
Coated chippings for hot rolled asphalt	20

NOTE 1 For some polymer-modified binders, several washings may be necessary to dissolve all the polymer.

NOTE 2 Long extraction periods may cause damage to the aggregate.

D.3.2.2.5 Centrifuge extractor method

The apparatus and procedure for the centrifuge extractor method are given in B.1.5.

NOTE 1 It is recommended to verify that the binder is well dissolved. If not, the minimum extraction time in Table B.1 should be increased.

NOTE 2 Toluene gives generally more accurate and more reproducible results than dichloromethane.

NOTE 3 EVA modified binders are generally very difficult to dissolve in a cold solution.

D.3.2.2.6 Cold mix dissolution of bitumen by agitation

The apparatus and procedure for cold mix dissolution of modified binder by agitation are given in B.1.6, except that the container should be shaken for at least 45 min instead of 30 min. Before starting, check the solubility of the modified binder in perchloroethylene, according to EN 12592. This method cannot be applied if the solubility of the modified binder is insufficient.

D.3.3 Separation of mineral matter

D.3.3.1 General

The apparatus and procedures to be used for the separation of the mineral matter are described in 5.3.1 and 5.3.2. Special conditions related to the different methods are recommended for mixtures containing polymer-modified binders. They are described in D.3.3.2 to D.3.3.5.

D.3.3.2 Continuous flow centrifuge

The requirements and procedure for separation of mineral matter by the continuous flow centrifuge are given in B.2.1, except that re-washing of the filler collected in the centrifuge cup should be repeated once more, after the decanted solvent has become virtually colourless.

NOTE A loss of mineral material is possible during centrifugation with the continuous flow centrifuge leading to an overestimation of the binder content.

D.3.3.3 Pressure filter

This method is not recommended for polymer-modified binder, because of risks of clogging.

D.3.3.4 Bucket centrifuge type 1

The requirements and procedure for separation of mineral matter by the bucket centrifuge type 1 method are given in B.2.3.

D.3.3.5 Bucket centrifuge type 2

The requirements and procedure for separation of mineral matter by the bucket centrifuge type 2 method are given in B.2.4

D.3.4 Binder quantity

For the determination of the binder quantity, follow the procedure as given in 5.4.2. If the soluble modified binder is not needed for further purposes, the difference method is the reference method. It is however recommended to use both methods as an initial check. Either method may be used subsequently, if the difference is 0,3 % or less.

D.3.5 Calculation and expression of results

Calculation and expression of results is performed according to 5.5.

D.4 Drying to constant mass

The requirements for drying to constant mass are the same as given in Clause 6.

D.5 Reporting of results

The reporting of results is as given in Clause 7. It is recommended to add the solvent used and the results of the solubility test.

D.6 Precision data

No reproducibility data are available.

Data are available of a repeatability experiment performed on a laboratory prepared bulk mix SMA-6.3 containing 6,6 % SBS modified binder. The rolling bottle machine was used for 1,5 h to dissolve the binder. The continuous flow centrifuge was used for extraction and the binder content was determined by difference. The repeatability (r) for a 95 % confidence level was found very comparable as in the case of unmodified binders: 0,1 % in the case of toluene as solvent and 0,2 % in the case of dichloromethane.

NOTE 1 No differences in the precision of the method are to be expected for polymer-modified binders compared to bitumens, insofar that the solvent being used was able to dissolve the polymer-modified binder completely.

NOTE 2 In case of insufficient solubility of the polymer, incorrect results are obtained and larger discrepancies between the different methods and different solvents are to be expected compared to bitumens.

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[1] EN 12592, *Bitumen and bituminous binders — Determination of solubility*

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[4] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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