BRITISH STANDARD

BS 5284:1993

Methods of

Sampling and testing mastic asphalt used in building and civil engineering

Confirmed
July 2011

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by Technical Committee B/546, Flexible sheets for water and water vapour control, to Subcommittee B/546/7, Asphalt and pitch mastic for building purposes, upon which the following bodies were represented:

Bitumen Modifers Association
Department of the Environment (Building Research Establishment)
Department of the Environment (Property Services Agency)
Federation of Civil Engineering Contractors
Low Temperature Coal Distillers Association of Great Britain Ltd.
Mastic Asphalt Council and Employers Federation
Mastic Asphalt Producers Association
Royal Institute of British Architects
Society of Chemical Industry

This British Standard, having been prepared under the direction of Technical Committee B/546, was published under the authority of the Standards Board and comes into effect on 15 August 1993

The following BSI references relate to the work on this standard:
Committee reference B/546/7
Draft for comment 89/12062 DC

ISBN 0 580 21546 6

Amendments issued since publication

Amd. No.	Date	Comments

Contents

Foreword

This British Standard has been prepared under the direction of Technical Committee B/546, Flexible sheets for water and water vapour control, and gives methods for sampling and testing of mastic asphalt used in building. It supersedes BS 5284:1976 which is withdrawn.

In this edition reference to pitch mastic has been deleted as this material is no longer in general use. The method for determination of water content and the funnel method for binder content and grading of mineral aggregate have been omitted. Where practicable, the methods have been aligned with those of Parts 100, 101 and 102 of BS 598 which deals with sampling and testing of bituminous materials for roads and other paved areas. Some mastic asphalts are used for both road and building applications. In such cases, whilst the methods of sampling may vary, it is not intended that different test methods should be specified.

WARNING. The methods described in this British Standard require the use of dichloromethane. This solvent is hazardous to health and is subject to occupational exposure limits as detailed in the latest edition of Guidance Note EH/40 [1] published by the Health and Safety Executive. Exposure levels are related to both handling procedures and ventilation provision and it is emphasized that adequate training should be given to staff employed in the usage of this substance.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 18, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This British Standard specifies methods for sampling and testing mastic asphalt used in building and civil engineering.

NOTE Attention is drawn to the safety recommendations given in Annex A.

2 References

2.1 Normative references

This British Standard incorporates, by reference, provisions from specific editions of other publications. These normative references are cited at the appropriate points in the text and the publications are listed on the inside back cover. Subsequent amendments to, or revisions of, any of these publications apply to this standard only when incorporated in it by updating or revision.

2.2 Informative references

This British Standard refers to other publications that provide information or guidance. Editions of these publications current at the time of issue of this standard are listed on the inside back cover, but reference should be made to latest editions.

3 Definitions

For the purposes of this British Standard the following definitions apply.

3.1
increment

portion of material taken by a single operation

3.2
bulk sample

combination of increments taken from a mass of material which the bulk sample is intended to represent

3.3
laboratory sample

whole or representative part of a bulk sample that is sent to a laboratory for testing

3.4
repeatability

quantitative expression of the random error associated with a single test operator in a given laboratory obtaining successive results with the same apparatus under constant operating conditions on identical test material. It is defined as that difference between two such single results as would be exceeded in the long run in only one case in twenty in the normal and correct operation of the test method

3.5 reproducibility

quantitative expression of the random error associated with test operators working in different laboratories, each obtaining single results on identical test material when applying the same method. It is defined as that difference between two such single and independent results as would be exceeded in the long run in only one case in twenty in the normal and correct operation of the test method

4 Methods of sampling

4.1 General principles

NOTE 1 Samples of mastic asphalt are normally required for one or more of the following three purposes.

a) Assessment of the constituent proportions of the material for quality control.

b) Judgement of conformance to the specification for the material.

c) Establishment of a specific property of a defined quantity or area of material.

Before any sampling is undertaken, the purpose shall be considered and an appropriate sampling procedure chosen.

If requested, all parties having a direct interest in the results of the tests on materials taken on site shall be informed when and where samples are to be taken so as to afford them the opportunity of witnessing the procedure. Samples shall be made available for the manufacturer's own testing.

NOTE 2 If all the details of the specified sampling procedure are not carried out, the results of the test may not be accepted as being representative of the material being sampled.

The size of the bulk sample shall be not less than that stated in the procedures given in **4.2** to **4.6** and, if so required, shall be large enough to provide laboratory samples for each of the interested parties.

4.2 Sampling during discharge at the manufacturing plant

Use a clean, rigid metal container for sampling the molten material. The container shall be of dimensions capable of receiving a 2 kg increment.

Take at least three increments.

Take each increment by placing the empty container in the stream of material being discharged from the mixer. Discard the increment if the container becomes surcharged during this procedure. Record the temperature of the mastic asphalt.

In the case of batch mixers, carry out the sampling while the mixer contains between one-third and two-thirds of the original charge. Thoroughly mix the increments together in a clean container while still molten to form a bulk sample of sufficient mass to provide laboratory samples having a mass not less than that given in Table 1.

4.3 Sampling of block material

Select at random and break not less than six blocks for every 10 t of material or part thereof.

Unless contamination is the subject of investigation, take increments of approximately equal size from the inner part of each block and combine to form a bulk sample of sufficient mass to provide laboratory samples having a mass not less than that given in Table 1.

Table 1 — Minimum mass of laboratory samples

Maximum size of coarse aggregate specified	Minimum number of increments	Minimum mass of each increment kg	Minimum mass of laboratory sample kg
Passing 3.35 mm sieve conforming to BS 410:1986	3	1	3
Passing 14.0 mm sieve conforming to BS 410:1986	3	2	6

If contamination is the subject of investigation, submit the whole block for examination.

4.4 Sampling from site remelting equipment and mobile mixers

Preferably carry out the sampling while the mixer contains between one-third and two-thirds of the original charge and the hand-stirred cauldron approximately one-half of the original charge. However, if this is not possible, record with the sample identification, the actual conditions, how much of the original charge remained and if fresh block material had been added prior to sampling.

Use a clean, rigid metal container for sampling the molten material. The container shall be of dimensions capable of receiving an increment of at least 2 kg.

Take at least three increments. Where sampling from a mixer, take each increment by placing the empty container in the stream of material being discharged. Discard the increment if the container becomes surcharged during this procedure. When taking the increment from the cauldrons, thoroughly stir the molten material immediately beforehand.

Thoroughly mix the increments together in a clean container while still molten to form a bulk sample of sufficient mass to provide laboratory samples having a mass not less than that given in Table 1.

4.5 Sampling from laid and finished material

Obtain an individual sample from laid and finished mastic using one of the following procedures.

a) Warm the perimeter of the sample area by application of molten mastic asphalt.

NOTE 1 The supply of molten mastic asphalt, prepared to restore the surface once the sample has been removed, may be used for this purpose.

With a knife or similar tool, cut through the total thickness of the material and carefully extract the sample.

NOTE 2 This method reduces damage to both the sample and the surrounding material to a minimum.

b) Cut out the sample using a mechanical cutting disc.

Do not use a blow torch for warming the material or cut the material cold by using a hammer and chisel as this could cause fracture of surrounding material.

When cutting out the sample note any factors which might influence the investigation.

NOTE 3 Such factors include surface finishes and blemishes, underlays, vent pipes and surface contamination.

An individual sample shall be of not less than 300 mm × 300 mm and, within this, a minimum area of 200 mm × 200 mm shall remain free from the molten mastic asphalt. Such a sample shall be regarded as representing only a limited area of the material.

For representative sampling of laid and finished mastic asphalt, select individual samples at the rate of not less than one for each 50 m^2 and part thereof of the area concerned, but in no case take less than three samples, distributed approximately equally over the area and not less than 0.5 m from a joint or the edge of a bay.

For the investigation of suspected faults or irregularities in material or workmanship take specific samples from the area(s) in question. Such samples shall be regarded as separate from those that have been selected for any sample intended to be representative of the work as a whole.

4.6 Size of sample

The bulk sample(s) taken in accordance with **4.2**, **4.3** or **4.4** shall be of sufficient mass to provide the required number of laboratory samples.

NOTE The minimum mass of laboratory samples is dependent on the maximum size of coarse aggregate specified for the mastic asphalt (see Table 1).

4.7 Information to be supplied with samples

Ensure that the sample is adequately identified with provision for reference to a schedule giving information on the appropriate items from the following:

a) location of mixing plant or site where laid;

b) name and address of authority giving instructions for the examination to be carried out;

c) sample identification;

d) type of material;

e) specification with which the material is intended to comply;

f) date of mixing or laying;

g) date of sampling (see note);

h) positions from which sample of laid material was taken;

i) number and nominal thicknesses of courses;

j) nature and condition of base;

k) nature of surface treatment (if any);

l) test to be made, or information sought;

m) name of sampler (in block capitals), signature and name of sampling organization.

To facilitate the testing procedure and the interpretation of test results give as much information as possible to the laboratory. Include site observations such as those noted when sampling laid and finished material in **4.5**.

NOTE Sampling should be within 24 h of laying for determinations intended to be recorded as the hardness number at the time of laying.

5 Preparatory laboratory treatment of sample

5.1 Preliminary inspection

Inspect the sample as soon as possible after receipt and note its condition.

5.2 Sample reduction and preparation

Unless the sample has been received at a temperature sufficient to enable the material to be broken down to an appropriate size for analysis, i.e. not larger than 20 mm, place the remainder of the sample in an oven at a temperature not exceeding 180 °C and, as soon as it is softened just sufficiently to be broken, remove it from the oven. Avoid prolonged heating.

Break the sample into small pieces and obtain a representative portion by taking pieces at random.

5.3 Preparation for hardness number test

Reconstitute a further representative portion of the sample by careful heating for the shortest possible time at a temperature not exceeding 205 °C.

Cast the molten material into duplicate hardness number test moulds and float finish. Avoid excessive floating as this can influence the hardness number.

The hardness number test mould shall be not less than 100 mm in diameter and not less than 25 mm in depth.

6 Hardness number test

NOTE The hardness number test should be used as follows.

a) *At the place of manufacture.* For quality control purposes as required by the appropriate standard.

b) *At the remelting and laying stage.* For quality control purposes as required by the appropriate standard.

c) *Laid material.* Laid material is contaminated with surfacing and underlay materials and there is no provision for the application of this test other than at the manufacturing and laying stages.

The stability of mastic asphalt increases with time so, depending on the age of the asphalt, hardness numbers on samples taken at a later date may therefore be significantly lower than at the time of laying. Most of the process is completed in the first year, and extensive investigations of this characteristic show that this can be equal to a 40 % reduction of the hardness number from that at the time of laying.

6.1 Principle

A flat-ended indentor pin in the form of a steel rod 6.35 mm in diameter is allowed to indent the mastic under a force of 311 N applied for 60 s whilst the mastic is maintained at the temperature specified in the appropriate standard. The depth of indentation is measured.

6.2 Apparatus

The apparatus, of which a suitable form is illustrated in Figure 1, and with which the force is conveniently applied by means of a lever giving a mechanical advantage, shall be capable of realizing the principle given in **6.1**.

6.3 Method

Cool the hardness number test piece, prepared in accordance with **5.3**, in air for not less than 3 h or in cold water for not less than 1 h.

Immerse the test piece in water, maintained to within ± 0.1 °C of the specified test temperature, for not less than 1 h prior to testing. Transfer the test piece to the test apparatus (see Figure 1) where it shall be again immersed in water, maintained to within ± 0.1 °C of the specified test temperature, throughout the test period.

Adjust the indentor pin so that it is lightly but firmly in contact with the sample, the pressure being no greater than is necessary to prevent lateral movement of the test piece.

Apply the specified force for 60 s and record the depth of the indentation at the end of this period to an accuracy of 0.1 mm.

Take at least five readings at test points not less than 25 mm apart and not less than 25 mm from the edge of each test piece.

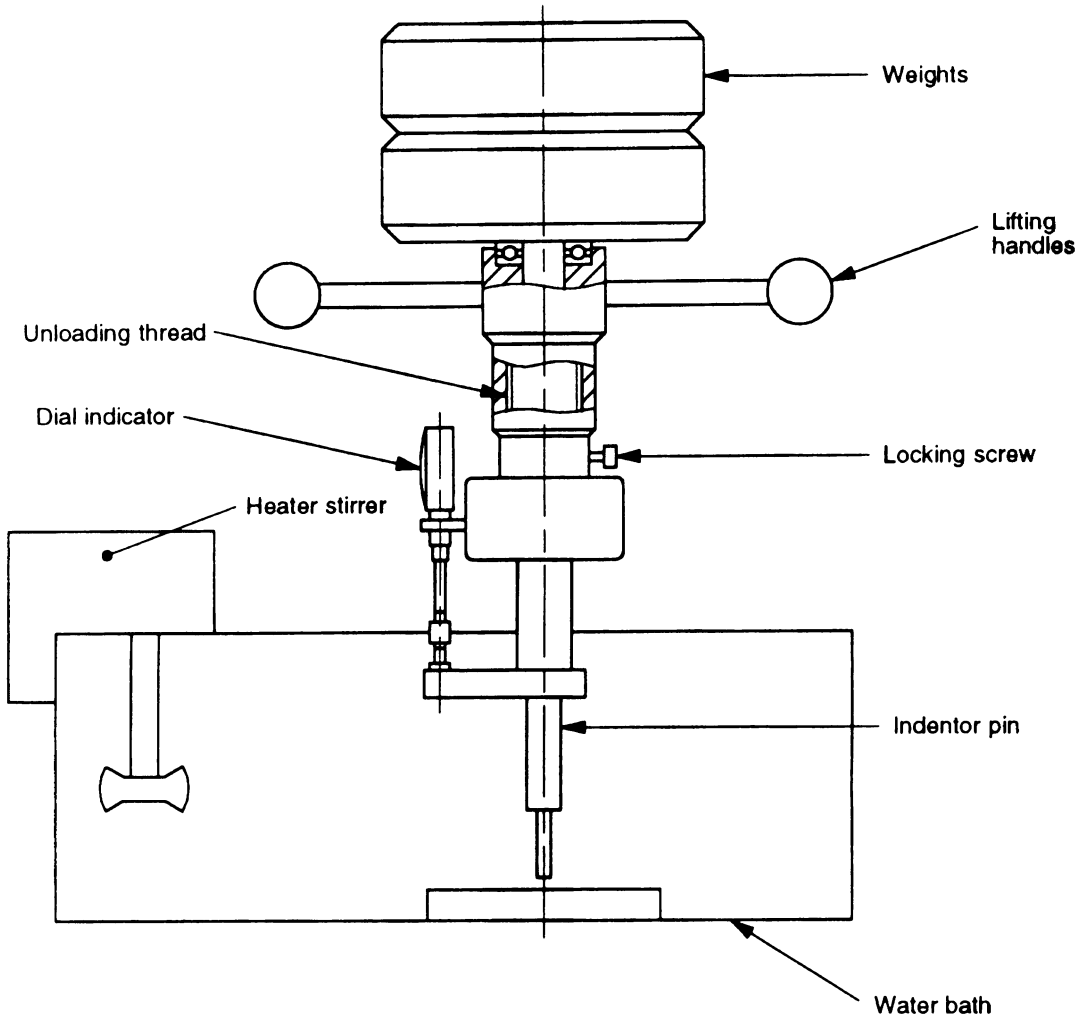


Figure 1 — A type of apparatus suitable for hardness testing

6.4 Expression of results

6.4.1 *Results*

Calculate the mean of the five test results and compare with the individual results. Reject any result that differs from the calculated mean by more than 2 hardness units (0.2 mm) or 15 % of the mean, whichever is the greater. If more than one result is rejected discard the specimen and test the duplicate specimen. If the test on the duplicate specimen fails to provide four acceptable results repeat the test on freshly cast specimens.

6.4.2 *Reporting of results*

Report the mean of the acceptable test results (to the nearest 0.1 mm).

7 Determination of binder content and grading of mineral aggregate

7.1 General

The binder content and grading of mineral aggregate shall be determined by one of the following methods:

a) the extraction bottle method (binder directly determined) in accordance with **7.2**;

b) the sieving extractor method in accordance with **7.3**.

NOTE The advantages and disadvantages of these methods depend on the circumstances of a particular case, and the choice of method is a matter for individual assessment. Nevertheless both methods, when carried out by a skilled operator, will produce comparable test results in all materials submitted for investigation provided that:

a) strict attention is paid to the details of the test procedure;

b) the operator has shown that he can achieve the repeatability requirements given in **7.5**.

7.2 Extraction bottle method (binder directly determined)

7.2.1 *Size of sample for analysis*

The mass of the sample for analysis shall be as given in Table 2.

7.2.2 *Apparatus*

NOTE Apparatus should be calibrated and traceable as recommended in Annex B.

7.2.2.1 *Metal bottles,* of appropriate capacity to the size of sample being analysed, e.g. 600 ml, 2 500 ml and 7 000 ml, with wide mouths and closures.

NOTE Bottles should not be charged more than 75 % of their capacity.

7.2.2.2 *Machine,* which will rotate the bottles at a speed of approximately 10 r/min to 30 r/min about their longitudinal axes.

7.2.2.3 *Volumetric flasks,* of appropriate capacity, e.g. 250 ml, 500 ml, 1 000 ml and 2 000 ml calibrated to at least class B accuracy of BS 1792:1982 or other system of measuring solvent calibrated as accurately as class B of BS 1792:1982.

NOTE 1 See also **B.5.2**.
NOTE 2 In the procedure, for clarity, reference to volumetric flasks only is made but this does not preclude the use of other systems of solvent measurement providing they fulfil the accuracy requirements.

7.2.2.4 *Centrifuge,* conforming to BS 4402:1982, capable of developing a relative centrifugal force (r.c.f.) of about 2 500 *g* calculated in accordance with the following formula:

$$\text{r.c.f.} = 1.12n^2r \times 10^{-6}$$

where

n is the number of revolutions per minute;

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

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

NOTE 1 A typical centrifuge suitable for this method carries two or more buckets fitted with centrifuge tubes of 50 ml capacity.
NOTE 2 It is strongly recommended that the speed of rotation should be verified regularly to ensure that the centrifuge maintains its performance at all times. The centrifuge should be maintained in accordance with Appendix B of BS 4402:1982.

7.2.2.5 *Filtration apparatus,* (see Figure 2) comprising a metal bottle such as described in **7.2.2.1**, a porous filter thimble, tubing and a 50 ml burette. The porous filter shall be of porcelain, alumina or similar material, 90 mm long by 20 mm diameter and of 2 μm to 4 μm pore diameter. The filter is closed by sealing in, to within 5 mm of the bottom, a length of metal tubing, approximately 300 mm long by 5 mm bore, that passes through a supporting ring of cork or metal that is mounted just inside the open end of the filter (see Figure 3). The seal is effected by either of the following methods.

Table 2 — Size of sample, sieves and minimum volume of solvent to be used for analysis

Type of material	Sample size range	Recommended sieve aperture size		Minimum volume of solvent for sieving extractor
	g	mm	μm	ml
Natural rock asphalt powder	500 to 550	2.36	600, 212, 75	2 000
Natural rock mastic ungritted	300 to 325	3.35 or 2.36	600, 212, 75	2 000
Natural rock mastic 10 % to 25 % coarse aggregate	300 to 325	3.35	600, 212, 75	2 000
Natural rock mastic 25 % to 50 % coarse aggregate	500 to 550	2.36	600, 212, 75	2 000
Limestone mastic ungritted	300 to 325	3.35 or 2.36	600, 212, 75	2 000
Limestone mastic 10 % to 25 % coarse aggregate	400 to 450	3.35	600, 212, 75	2 000
Limestone mastic 25 % to 50 % coarse aggregate	500 to 550	2.36	600, 212, 75	2 000

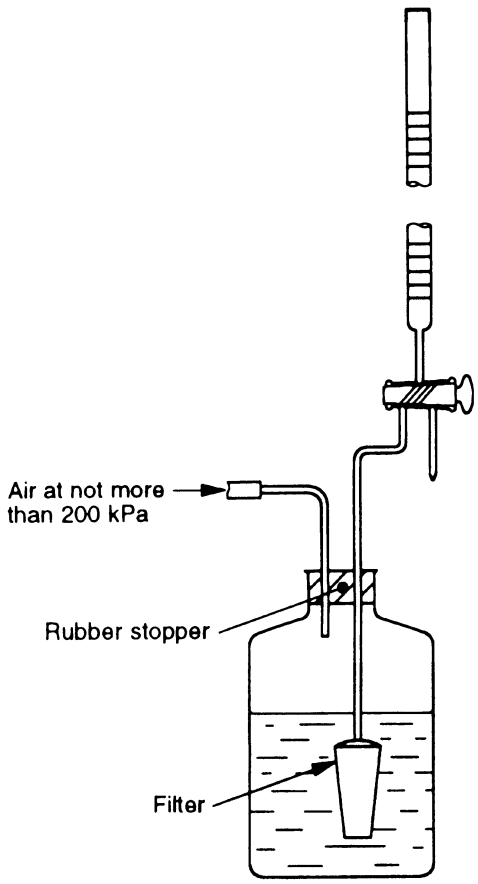


Figure 2 — Filtration apparatus

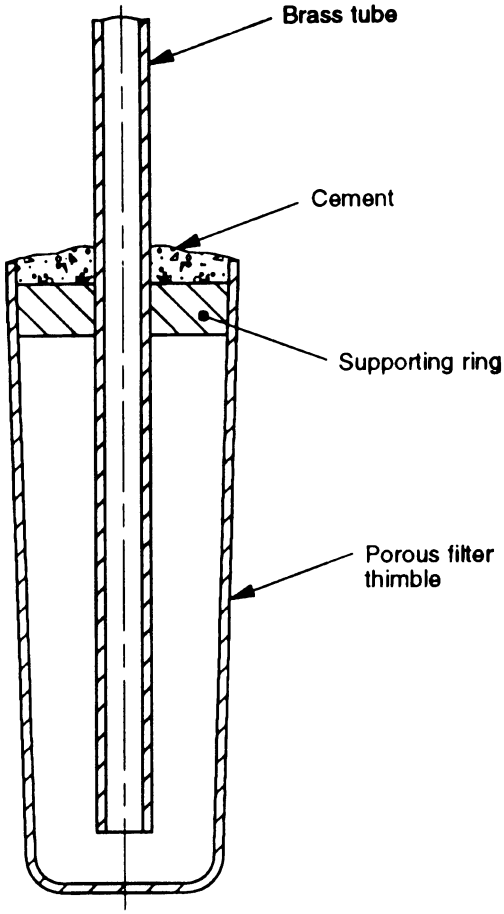


Figure 3 — Porous filter thimble

a) A cement paste composed of copper oxide powder, prepared by the direct oxidation of copper wire, of about 425 µm particle size, and phosphoric acid is used to seal the joint. The joint is left to dry out for a few hours.

b) Alternatively, a rubber stopper or sealing compound may be used.

Care shall be taken to ensure that the seal does not split the filter thimble.

7.2.2.6 *Recovery apparatus,* comprising a water bath, with an electric heater capable of maintaining boiling water in the bath throughout the recovery procedure, a flat-bottomed flask of 200 ml or 250 ml capacity, a vacuum gauge, a vacuum reservoir and a method of maintaining reduced pressure, e.g. a filter pump.

NOTE Figure 4 illustrates a typical recovery apparatus but this particular arrangement is not compulsory provided that the conditions detailed in **7.2.4**, for recovering the binder from the solution, are strictly followed.

7.2.2.7 *Pressure filter apparatus,* comprising a pressure filter of appropriate size (one taking a filter paper of 270 mm diameter is suitable), an air pump for supplying oil-free air at approximately 200 kPa and a funnel for supporting the sieves, and for insertion into the filling orifice in the pressure filter.

7.2.2.8 *Supply of suitable grade filter papers*[1)], to fit the pressure filter.

7.2.2.9 *Set of sieves,* conforming to BS 410:1986, of appropriate aperture sizes.

7.2.3 *Reagent*

7.2.3.1 *Dichloromethane* (methylene chloride), conforming to BS 1994:1953.

NOTE 1 See warning note in the foreword regarding the use of solvents.

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

[1)] No. 1 and No. 5 Whatman® filter papers are suitable. "Whatman" is the registered trademark of Whatman Paper Ltd.

7.2.4 *Method*

Weigh the sample to the nearest 0.05 % of the mass taken and introduce it into the metal bottle.

Provided the sample is cool to the touch add dichloromethane measured by means of volumetric flasks to the sample to give a solution of about 3 % (*m*/*gm*) concentration of soluble binder.

CAUTION. If the temperature of the sample is more than 3 °C above the solvent temperature, a dangerously high concentration of solvent vapour may be produced and pressure may build up in the bottle leading to the risk of explosion. Furthermore, the volume of solvent lost by evaporation may be sufficient to give an erroneously high soluble binder content for the sample.

NOTE 1 The required total volume of solvent, V (in ml), should be estimated from the following formula. The total volume should be corrected to the nearest 250 ml.

$$V = \frac{MS_E}{C_S}$$

where

M is the mass of the sample (in g);

S_E is the estimated percentage of soluble binder in the sample;

C_S is the required concentration of solution (in %).

Close the bottle and roll on the bottle rotating machine for 45 min except that where degradation of the aggregate is possible due to attrition in the rolling bottle, the rolling should be intermittent but the rolling time should be at least 15 min in total.

NOTE 2 For material which has been laid for a period the rolling time may need to be extended to ensure complete extraction of the soluble binder.

Free a portion of the binder solution from insoluble matter either by means of centrifuging for 20 min in completely closed tubes or by means of the filtration apparatus using a dry, binder-free filter.

NOTE 3 If filtration is prolonged, due to fine mineral matter being present in the binder solution, inaccurate results may be obtained and in this case centrifuging is essential.

Minimize the loss of solvent during the extraction and centrifuging or filtration procedures.

From this point carry out the procedure in duplicate.

NOTE 4 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 that between 0.75 g to 0.95 g of soluble binder is obtained.

Dry and weigh the dry flask to the nearest 0.01 g.

Measure a sufficient amount of the centrifuged solution into the flask, using the burette, to give a residue of 0.75 g to 0.95 g of soluble binder after evaporation of the solvent.

NOTE 5 An estimate of the volume of solution (aliquot portion), v (in ml) required is given by:

$$v = \frac{100\,V}{MS_E}$$

where

V is the total volume of solvent (in ml);

M is the mass of the sample (in g);

S_E is the estimated percentage of soluble binder in the sample.

The estimate of the volume shall be rounded to the nearest 5 ml.

Remove the solvent from the binder solution by connecting the flask to the recovery apparatus, 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 with a rotary motion so that the binder is deposited in a thin layer on the walls of the flask. Do not allow pressure above atmospheric to develop in the flask during the evaporation of the solvent.

NOTE 6 It is recommended that the distillation be carried out under reduced pressure. If reduced pressure is used the pressure should be not less than 60 kPa.

NOTE 7 Two flasks may be connected to the vacuum system and the distillation may be carried out simultaneously provided that both are rotated continuously.

There is a relatively rapid reduction in pressure when the bulk of the solvent has been removed and at this stage frothing usually occurs.

When frothing occurs maintain the flask at approximately half its depth in the boiling water. Reduce the pressure to 10 kPa and maintain at this pressure for 5 min.

Remove the flask from the bath and admit air to the apparatus to increase the pressure to atmospheric. Wipe the flask dry and then disconnect it, care being taken to prevent the entry into the flask of any water that may have collected where the rubber stopper joins the flask. Remove the last traces of solvent that remain in the flask by a gentle current of clean, oil- and water-free air.

Cool the flask in a desiccator and weigh to the nearest 0.01 g. If the quantity of soluble binder recovered lies outside the range 0.75 g to 0.95 g repeat the recovery with another portion of the solution having the volume suitably adjusted.

If the difference between the duplicate recoveries is greater than 0.02 g reject these results and repeat the recovery of the binder in duplicate on further aliquot portions. Use the average of the duplicate determinations for calculation of the soluble binder content.

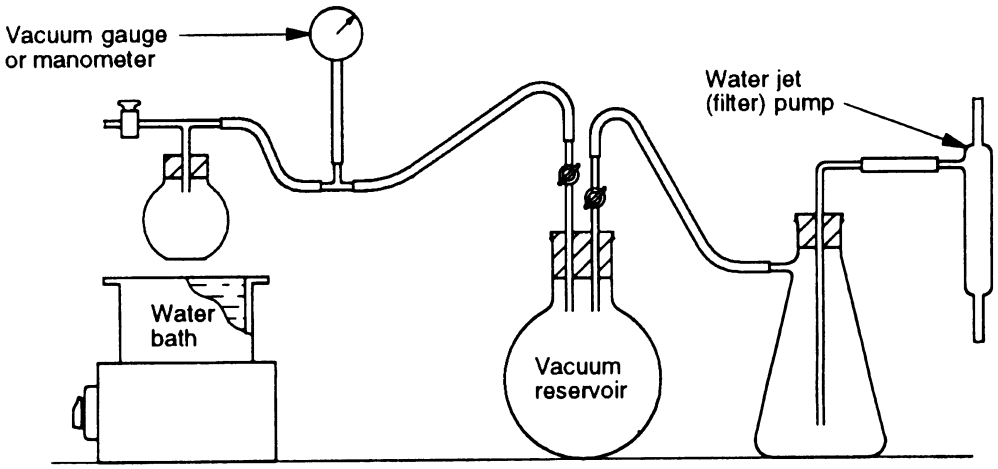


Figure 4 — Recovery apparatus showing necessary features

7.2.5 *Calculation of soluble binder content*

Calculate the soluble binder content S [in % (m/m)] on the dry sample by means of the following formula:

$$S = \frac{100zV}{vm}1 + \frac{z}{dv}$$

where

m is the mass of sample (in g);

z is the average mass of binder recovered from the two aliquot portions (in g);

V is the total volume of solvent (in ml);

v is the volume of each aliquot portion (in ml);

d is the density (in g/ml) (1.0 in the case of bitumen).

7.2.6 *Washing of mineral aggregate*

After removing sufficient solution for the determination of the soluble binder content, pour the liquid contents of the extraction bottle (including the fine matter in suspension but taking care not to carry over any other aggregate) through a nest of appropriate sieves and through the funnel into the pressure filter, which shall be fitted with a suitable grade of filter paper[2)]. Then force the liquid through the filter paper under pressure.

NOTE 1 For mixtures containing a high percentage of material passing the 75 µm sieve it may be advisable to superimpose a coarser grade filter paper[3)] on the filter paper fitted on the funnel to prevent clogging of the latter.

Shake the aggregate remaining in the bottle with a further quantity of solvent, i.e. about half the quantity of solvent used originally or about 1 000 ml, whichever is the smaller. Immediately after shaking pour the solution through the nest of sieves into the pressure filter, extreme care being taken to ensure no loss of mineral matter. Then force the solution through the pressure filter. Repeat this process until no discoloration of the dichloromethane is visible and the washings are visibly free from material in suspension. At this point carefully transfer the contents of the bottle on to the nest of sieves. Rinse the bottle once more to remove as much of the mineral matter as possible. Force the final washings through the pressure filter.

NOTE 2 Any mineral matter in the portion of solution removed for the determination of binder content should be recovered with the rest of the aggregate.

7.2.7 *Grading of mineral aggregate*

NOTE 1 Provided the aggregate was thoroughly washed, the proportion of material passing the 75 µm test sieve remaining with the aggregate should be small, i.e. less than 1.5 % of the total aggregate. If the amount of material passing the 75 µm test sieve remaining with the aggregate is greater than 1.5 % of the total aggregate, the result for the material passing the 75 µm test sieve may be incorrect and is indicative of insufficient washing.

Dry and grade the aggregate on the sieves and/or tray using the general principles described in BS 812-103 taking particular care to ensure that the sieves, especially the fine mesh sieves, are not overloaded. Where necessary separate the aggregate into two fractions:

a) coarse aggregate fraction, retained 3.35 mm or 2.36 mm sieve;

b) fine aggregate fraction, passing 3.35 mm or 2.36 mm sieve.

[2)] Whatman No. 5 filter paper is suitable.

[3)] Whatman No. 1 filter paper is suitable.

Note the masses of the two fractions.

Grade all of the coarse aggregate fraction on a nest of sieves appropriate for the mixture. For the grading of the fine aggregate fraction take a sample of 100 g to 200 g. When required split the fine aggregate fraction using a sample divider. Grade the fine aggregate on a nest of sieves of appropriate mesh sizes. When only a portion of the fine aggregate fraction is graded, multiply the masses of graded fine aggregate by the ratio of the total mass of the fine aggregate fraction to the mass of the portion taken.

Determine the mass of material passing the 75 µm sieve by either of the following methods.

a) Dry and weigh the material passing the 75 µm sieve recovered from the pressure filter. To obtain the total mass of the material passing the 75 µm sieve, add the mass of the material passing the 75 µm sieve from the grading to the mass of the material recovered from the pressure filter.

NOTE 2 The total mass will include the insoluble portion of the binder.

b) Alternatively, the aggregate passing the 75 µm sieve may be estimated by difference i.e:

$$m_p = m_s - (m_B + m_{NP})$$

where

m_p is the mass of material passing the 75 µm sieve (in g);

m_s is the mass of the sample (in g);

m_B is the mass of total binder (in g);

m_{NP} is the mass of aggregate material coarser than the 75 µm sieve (in g).

Calculate the grading of the total aggregate.

7.2.8 *Results*

Express the results as a percentage by mass and calculate to the nearest 0.1 % (m/m). The actual calculations carried out will depend upon the form in which the results are required for comparison with the specification for the mixture.

Report the binder content to the nearest 0.1 % (m/m) of the total dry sample.

Report the grading of the mineral aggregate to the nearest 0.1 % (m/m) of the total dry sample.

7.3 Sieving extractor method

7.3.1 *Size of sample for analysis*

The mass of the sample shall be as given in Table 2.

7.3.2 *Apparatus*

NOTE Apparatus should be calibrated and traceable as recommended in Annex B.

7.3.2.1 *Sieving extractor,* (see Figure 5) for simultaneous extraction of the binder and sieving of the aggregate, consisting of a metal cylindrical vessel 200 mm in diameter, having a domed top with an orifice which can be closed with a liquid-tight closure. The height of the vessel shall be at least 90 min.

NOTE 1 For samples containing aggregate retained on a 3.35 mm sieve a height of 120 mm is necessary.

The walls of the vessel shall be of double thickness when analysing samples containing aggregate of a size likely to damage it.

NOTE 2 The base of the vessel can be formed by mesh conforming to BS 410, e.g. mesh of 3.35 mm or 2.36 mm aperture size.

The cylindrical vessel is mounted above a nest of sieves of appropriate fine mesh sizes, the sieves being separated from adjacent sieves by suitable washers to prevent any leakage of solvent. The sieves are 200 mm in diameter and 25 mm deep (see Table 2).

By means of a clamping device the assembly is attached to a baseplate to which a rocking motion can be imparted by attachment to a flywheel and electric motor drive. The assembly rocks through an angle of approximately 22° at a frequency of 2 Hz. The baseplate is fitted with a draincock (see note).

NOTE An approximately 6 mm bore petrol draincock is suitable.

7.3.2.2 *Volumetric flasks,* of 250 ml, 500 ml, 1 000 ml and 2 000 ml capacity calibrated to at least class B accuracy of BS 1792:1982 or other systems measuring solvent calibrated as accurately as class B of BS 1792:1982.

NOTE See also **B.5.2**.

7.3.2.3 *Centrifuge,* as described in **7.2.2.4**.

7.3.2.4 *Filtration apparatus,* as described in **7.2.2.5**.

7.3.2.5 *Metal bottle,* of about 2 500 ml capacity fitted with a rubber stopper.

7.3.2.6 *Recovery apparatus,* as described in **7.2.2.6**.

7.3.2.7 *Set of sieves,* conforming to BS 410:1987, of appropriate aperture sizes.

7.3.3 *Reagent*

7.3.3.1 *Dichloromethane,* conforming to BS 1994:1953.

NOTE See warning note in the foreword regarding the use of solvents.

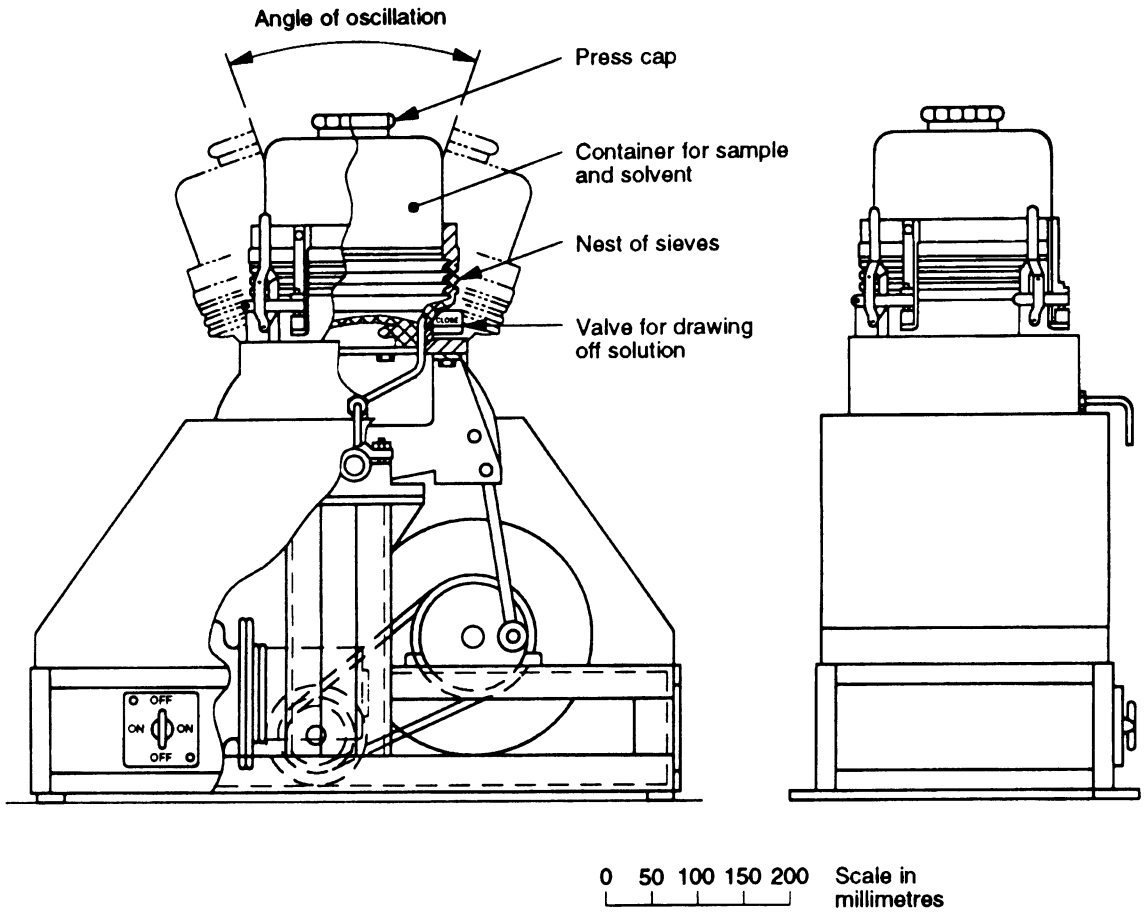


Figure 5 — Sieving extractor

7.3.4 ***Method***

Carry out the following procedure on each of the duplicate samples.

Weigh the sample to the nearest 0.1 g of the mass taken.

NOTE 1 If the sample has been heated to assist in sample reduction it is important that it should have been cooled to ambient air temperature before testing.

Collect the sieves necessary for the determination and inspect for damage and cleanliness. Assemble them on the base of the sieving extractor so that all joints are free from grit and so that they nest without locking together due to distortion or damage of the rims of the sieves. Adjust the clamps evenly so that no leakage of solvent through the joint is possible.

Add dichloromethane, measured by means of volumetric flasks, to the nest of sieves in the quantity given in Table 2.

NOTE 2 If sieves additional to those given in Table 2 are used the volume of solvent added should at least cover the mesh of the coarsest sieve.

NOTE 3 The difference between the temperature of the solvent and the temperature of the binder solution should not exceed 3 °C, to avoid any risk of build-up of solvent vapour.

NOTE 4 At ambient temperatures above 25 °C there will be an increased risk of inaccuracy due to evaporation.

When the joints of the sieves have been checked and found to be free from leaks, introduce the weighed sample onto the top sieve.

Close the orifice in the top by a liquidproof closure and set the filtration apparatus in motion. Continue shaking for normal ungritted and gritted mastic for 20 min, and in the case of heavily chipped mastic for 25 min.

NOTE 5 In some cases it may be found that the rate of extraction of binder is too slow for the extraction to be completed in the specified time. In such cases the periods of shaking should be broken by periods of soaking without agitation to maintain the total shaking time specified.

After shaking for the specified time replace the liquidproof closure by one which will allow entry of air whilst preventing spillage of solution.

Run the solution of binder in dichloromethane through the draincock into the metal bottle.

NOTE 6 During drainage, shaking may be continued for up to 5 min; if shaking is more prolonged the aggregate may degrade. Under certain conditions drainage may be very slow due to a liquid lock forming on the fine sieves and in this case drainage may be assisted by carefully blowing a small quantity of air through the sieves from below, via the draincock, and by continuing the shaking.

Free a portion of the binder solution from insoluble matter, either centrifuging for 20 min in completely closed tubes or by means of the filtration apparatus using a dry, binder-free filter. If filtration is prolonged, due to fine material matter being present in the binder solution, inaccurate results may be obtained and in this case centrifuging shall be carried out.

NOTE 7 The loss of solvent should be kept to a minimum during the extraction and centrifuging.

Weigh the flat-bottomed flask to the nearest 0.01 g.

Measure sufficient of the centrifuged or filtered solution into the flask, using the burette, to give a residue of 0.75 g to 0.95 g of total soluble binder after evaporation of the solvent.

NOTE 8 An estimate of the volume of solution (aliquot portion) required is given by:

$$\text{Aliquot portion } v\,(\text{ml}) = \frac{100\ V}{MS_E}$$

where

V is the total volume of solvent (in ml);

M is the mass of the sample (in g);

S_E is the estimated percentage of soluble binder in the sample.

The estimate of the volume shall be rounded to the nearest 5 ml. Remove the solvent from the binder solution by connecting the flask to the recovery apparatus, partially immersing the flask in the bath of boiling water and distilling off the solvent. While the distillation is proceeding shake the flask with a rotary motion so that the binder is deposited in a thin layer on the walls of the flask. Do not allow pressure above atmospheric to develop in the flask during the evaporation of the solvent.

NOTE 9 It is recommended that the distillation be carried out under reduced pressure. If reduced pressure is used the pressure should be not less than 60 kPa.

NOTE 10 Two flasks may be connected to the vacuum system and the distillation may be carried out simultaneously provided that both are rotated continuously.

There is a relatively rapid reduction in pressure when the bulk of the solvent has been removed and at this stage frothing usually occurs.

When frothing occurs, maintain the flask at approximately half its depth in the boiling water. Reduce the pressure to 10 kPa and maintain at this pressure for 5 min.

Remove the flask from the bath and admit air to the apparatus to increase the pressure to atmospheric. Wipe the flask dry and then disconnect it, care being taken to prevent the entry into the flask of any water that may have collected where the rubber stopper joins the flask. Remove the last traces of solvent that remain in the flask by a gentle current of air.

Cool the flask in a desiccator and weigh to the nearest 0.01 g. If the quantity of soluble binder recovered lies outside the range of 0.75 g to 0.95 g repeat the recovery with another portion of the solution having the volume slightly adjusted. Make duplicate recoveries and use the average result for calculation of the binder content.

7.3.5 *Calculation*

Calculate the soluble binder content, S [in % (m/m)], on the dry sample by means of the following formula:

$$S = \frac{100\ zV}{vm}\left(1 + \frac{z}{dv}\right)$$

where

m is the mass of undried sample (in g);

z is the average mass of binder recovered from the two aliquot portions (in g);

V is the total volume of solvent (in ml);

v is the volume of aliquot portion (in ml);

d is the density, (in g/ml) (1.0 in the case of bitumen);

Calculate the total binder content, B [in % (m/m)], on the dry sample by means of the following formula:

$$B = \frac{100\,S}{T}$$

where

S is the soluble binder content as previously calculated [in % (m/m)];

T is the mass of binder soluble in the solvent employed (in %).

7.3.6 *Grading of mineral aggregate*

Wash the aggregate remaining in the sieving extractor free from solution and aggregate passing the 75 µm sieve using several washes each of about 750 ml of dichloromethane.

NOTE The rinsing process will be assisted by shaking the sieves briefly.

Dismantle the apparatus, place the sieves on suitable trays and dry the aggregate in a ventilated cupboard. Grade the aggregate retained on the top sieve on appropriate sieves using the method described in BS 812-103. To ensure complete grading of the finer sieves, cover each sieve by a spare fine sieve and shake over a suitable receiver. Add any material that is collected in the receiver to the next finer sieve.

When grading has been completed check the sum of the masses of aggregate retained on the individual sieves by combining the graded aggregate and reweighing.

Calculate the amount of material passing the 75 µm sieve by difference as follows:

$$M_p = M_s - (M_B + M_{NP})$$

where

M_p is the mass of material passing the 75 µm sieve (in g);

M_s is the mass of the sample (in g);

M_B is the mass of total binder (in g);

M_{NP} is the mass of aggregate material coarser than the 75 µm sieve (in g).

7.4 Wet sieving by decantation method for fine powders

7.4.1 *Apparatus*

7.4.1.1 *Scale or balance,* of capacity not less than 250 g, accurate to 0.001 g.

7.4.1.2 *Heated fume cupboard,* thermostatically controlled to maintain a temperature of 105 °C ± 5 °C.

7.4.1.3 *Test sieves,* conforming to BS 410:1986, having the following mesh sizes: 75 µm, 212 µm, 600 µm and 2.36 mm. The sieves to be 25 mm deep and 200 mm in diameter.

7.4.1.4 *Non-aluminium metal beaker,* having a capacity of 1.2 l.

7.4.1.5 *Metal trays.*

7.4.1.6 *Metal stand and metal bowl*, of capacity 10 l.

7.4.2 *Reagent*

7.4.2.1 *Dichloromethane,* conforming to BS 1994:1953.

NOTE See warning in foreword regarding the use of solvents.

7.4.3 *Procedure*

Dry the test sample in a shallow metal tray in the fume cupboard for 24 h ± 0.5 h at a temperature of 105 °C ± 5 °C, cool and weigh.

Place the sample (approximately 100 g) in the metal beaker and cover with 500 ml of dichloromethane. Vigorously stir the contents of the beaker and pour the fines, in suspension, into the nested sieves. Arrange the sieves with the coarser sieves at the top and support by the metal stand placed in the 10 l metal bowl. Repeat the decanting procedure four times using 500 ml dichloromethane for each washing.

Finally, wash the aggregate remaining in the beaker completely into the nest of sieves by means of a wash bottle. Carefully place the 2.36 mm, 600 µm and 212 µm mesh sieves on clean metal trays and place in the fume cupboard under infra-red lamps to dry. Use a final 500 ml of dichloromethane to wash the powder remaining on the 75 µm mesh sieve, before placing the sieve and contents in the fume cupboard to dry.

When judged to be dry, shake each sieve separately until not more than a trace of material passes. On completion of sieving weigh the material retained on each sieve.

7.5 Repeatability and reproducibility

7.5.1 *Repeatability (r)*

The identical test material for repeatability tests shall be obtained by dividing a sample of twice the size required for a single test by the sample reduction procedure described in **5.2**.

7.5.2 *Reproducibility (R)*

The identical test material shall be obtained by first dividing a sample of eight times the size required for analysis into two approximately equal portions, one for each laboratory and then each laboratory shall reduce its portion to the size required for a single test by the sample reduction procedure described in **5.2**.

7.5.3 *Limits for repeatability and reproducibility*

The limits for the difference between the analysis results on two samples obtained from the same bulk sample shall be as given in Table 3. The two samples shall have been obtained by the sample reduction procedure described in **5.2**.

7.5.4 *Systematic differences in the results of analyses obtained by two laboratories*

NOTE 1 Differences in the results of analyses carried out in two laboratories, on test samples obtained from the same bulk sample may be due to errors in the sample reduction and/or the analysis procedures adopted by the analyst.

Any systematic difference in the comparison of groups of test results shall be investigated to detect the cause or causes of the bias.

NOTE 2 This bias will generally be due to non-adherence to the detail of the method of analysis if the same test method is being used or, in isolated cases, small differences may be due to the particular method of test selected. If differences arise when the same test method is being used it will be possible, with co-operation between the laboratories, to eliminate the cause.

If small differences occur, due to test methods being different, the interpretation of the test results, with respect to the specification requirements, shall be examined by the two laboratories concerned at the earliest opportunity.

Table 3 — Limits for repeatability and reproducibility of a sample obtained from the same bulk sample

Constituent	Repeatability %	Reproducibility %
Binder content:		
calculated on the mortar for mastic asphalt	0.4	0.6
Coarse aggregate content:		
retained on 600 µm sieve	5.0	7.0

8 Recovery of bituminous binders

NOTE This method describes a procedure for recovering the binder from mastic asphalt in a form suitable for further testing. The procedure is suitable for the recovery of bitumen of penetration grade.

The separation and recovery are designed to cause the minimum change in the properties of the binder.

It should be noted that the properties of the soluble bitumen recovered will not be the same as those of the total binder when recovered from mixtures containing lake asphalt.

8.1 Principle

8.1.1 The binder is separated from the sample by dissolving in dichloromethane. After removal of undissolved solids the solution is concentrated by atmospheric distillation through a fractionating column. The last traces of solvent are removed from the concentrate by distillation at a temperature of 175 °C, or 100 °C above the expected softening point, with the aid of a stream of carbon dioxide gas, and the pressure reduced to 20 kPa.

8.2 Apparatus

8.2.1 *Container,* in which the sample and solvent can be agitated together.

8.2.2 *Filtration apparatus,* comprising the following:

a) 200 mm diameter funnels;

b) 320 mm diameter filter papers[4)] of suitable grade;

c) 225 mm diameter cover plates;

d) suitable receivers for the filtered solution, e.g. chemical bottles of capacity 2.5 l;

e) centrifuge, conforming to BS 4402:1982, capable of developing a relative centrifugal force (r.c.f.) of 2 500 *g* calculated in accordance with the following formula:

$$\text{r.c.f.} = 1.12n^2r \times 10^{-6}$$

where

n is the number of revolutions per minute;

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

8.2.3 *Distillation apparatus,* as illustrated in Figure 6, the essential parts of which are as follows:

a) *500 ml round-bottomed flask,* of heat-resisting glass fitted with a three-necked adaptor. The central neck is used either to accommodate a glass link stirrer (as shown in Figure 7) or a glass tube of 4 mm to 6 mm internal diameter for sweeping carbon dioxide through the flask when required. To one side-neck is fitted a 250 ml stoppered separating funnel the stem of which is elongated into a tip extended well into the neck as illustrated in Figure 7. The other side-neck is connected to the fractionating column followed by an efficient water-cooled glass condenser and receiving system. The fractionating column is of the Dufton or Vigreux type, having an effective length of 300 mm to 400 mm and may be vacuum-jacketed.

NOTE It is important to ensure that there is no constriction in the column which can accelerate, even locally, the rate of passage of vapours because if this occurs flux oils may be dispersed as a mist and carried over into the receiver.

The receiver system includes a tap by which the main receiver can be isolated from the condenser.

All connections shall be made by means of ground-glass joints.

b) *Filter pump and gauge,* indicating pressure from approximately 10 kPa to 100 kPa.

c) *Oil bath,* electrically heated, with adequate power to raise the temperature of the oil to 175 °C, or 100 °C above the softening point of the recovered bitumen.

d) *Jack,* for raising or lowering the oil bath.

e) *Flowmeter,* having a range of 0 ml to 30 ml free flow of carbon dioxide ($C0_2$) per minute at 15 °C and 20 kPa pressure.

f) *Thermometer,* capable of reading 0 °C to 200 °C.

[4)] Whatman® No. 5 filter paper is suitable.

8.3 Reagents

8.3.1 *Dichloromethane,* conforming to BS 1994:1953.

NOTE See warning in the foreword regarding the use of solvents.

8.3.2 *Calcium chloride,* fused.

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

8.4 Procedure

8.4.1 *Preparation and extraction of the sample*

Place sufficient of the sample to contain approximately 250 g soluble binder in the container (see **8.2.1**). Add approximately 1 500 ml of dichloromethane and sufficient calcium chloride to absorb any water present in the sample. Agitate the contents of the container until all of the soluble binder has dissolved.

8.4.2 *Clarification of binder solution*

Allow the solution to stand for approximately 10 min and remove the insoluble material by filtration or centrifuging, or a combination of both, e.g. filtration overnight followed by completion of the separation next morning by centrifuging.

NOTE Centrifuging is essential when fine mineral matter is present in the sample.

If centrifuging is used, maintain the speed of the centrifuge at a level that ensures a relative centrifugal force of at least 2 500 *g* for a minimum of 20 min.

During the separation make every effort to prevent any moisture, due to condensation, entering the solution.

8.4.3 *Checking the apparatus for air leaks*

Assemble the glassware and connect together, in sequence, the carbon dioxide cylinder, gas regulator, flowmeter and gas delivery tube. Insert the gas delivery tube into the three-necked adaptor. With the carbon dioxide cylinder valve closed and all other valves and glass taps suitably adjusted, reduce the pressure in the whole of the apparatus to 20 kPa. Isolate the apparatus from the source of the reduced pressure and check that there is no increase in the pressure in the apparatus.

NOTE It is most important that no air is allowed to enter the apparatus through the carbon dioxide supply system, i.e. through the regulator and flowmeter unions and valves, and the flexible tubing connections. If a needle valve is fitted to the outlet of the gas regulator a pressure above atmospheric can be maintained on the gas side of the needle valve thus preventing the ingress of air.

8.4.4 *Distillation procedure*

Replace the gas delivery tube by the link stirrer and add to the flask two or three pieces of porous pot, or similar material, to prevent bumping.

NOTE Ensure that the size of material used to prevent bumping does not obstruct the insertion of the gas delivery tube or the flow of carbon dioxide.

Introduce 100 ml of solution (see **8.5.2**) into the distillation flask through the separating funnel. Agitate the solution by revolving the link stirrer at approximately 4 rev/s and raise the temperature of the oil bath to approximately 100 °C. When distillation starts slowly introduce further solution into the flask, keeping the volume in the flask at a minimum. In no case shall the volume exceed 250 ml. When all the solution has been added to the flask allow the contents to concentrate. During concentration allow the temperature of the oil bath to gradually increase without adjusting the heat input.

When the rate of distillation has slackened to approximately 10 drops per minute increase the temperature of the oil bath over a period of 20 min to 30 min to 175 °C, or 100 °C above the expected softening point of the binder, whichever is the greater. If foam forms during concentration it may be advantageous to keep stopping and starting the stirrer.

After the rate of distillation has dropped to 3 drops per minute or 4 drops per minute for 5 min consecutively adjust the oil bath so that the oil level is 10 mm to 20 mm above the liquid level in the flask. Replace the separating funnel by a glass stopper and the link stirrer by the gas delivery tube which should reach to within 5 mm of the bottom of the flask. Empty the receiver.

Pass carbon dioxide through the residue in the flask at 10 ml/min and gradually reduce the pressure in the apparatus over a period of 10 min to 15 min until the pressure has fallen to 20 kPa. During the reduction of the pressure maintain the flow of carbon dioxide at 10 ml/min by suitable adjustment of the control valve. Maintain the bath temperature, pressure and flow of carbon dioxide for 45 min.

Isolate the apparatus from the sources of heat and reduced pressure. Allow the pressure within the apparatus to increase to atmospheric by the ingress of carbon dioxide at 10 ml/min.

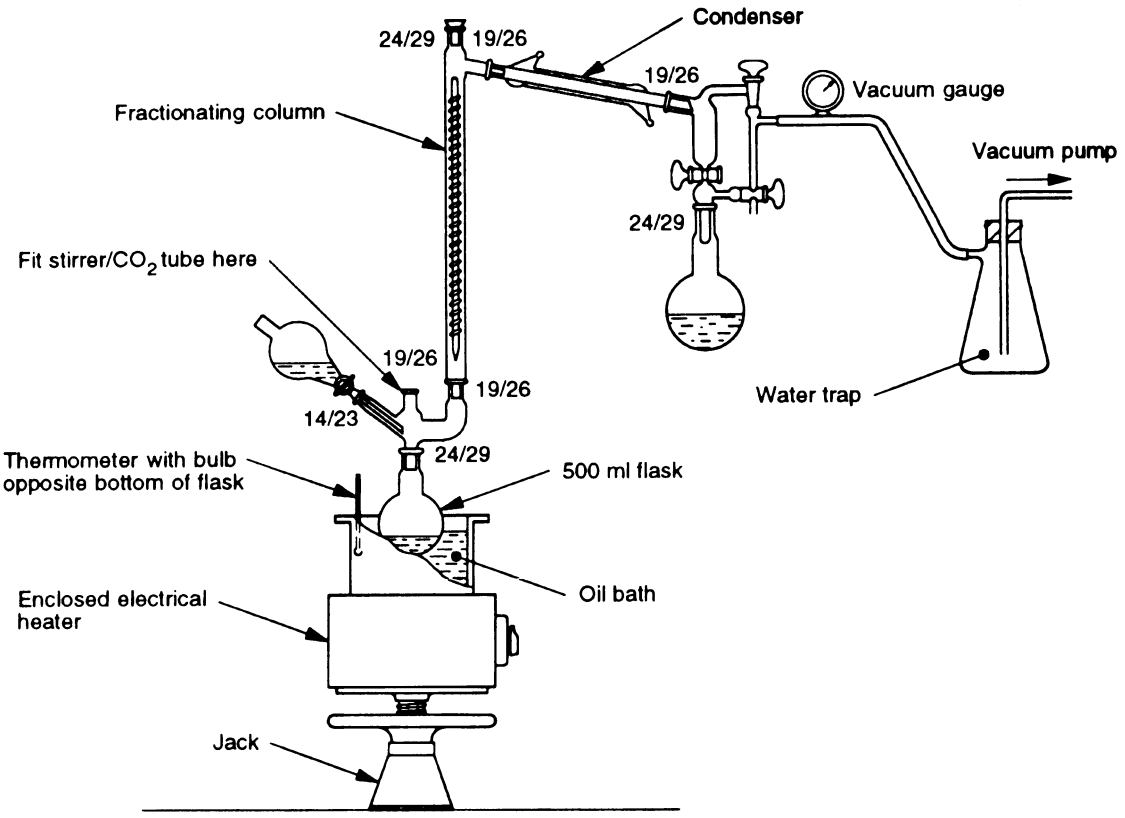
Allow the fractionating column to drain and the contents of the flask to cool below their fuming point. Remove the flask and swift to mix the contents.

The sample is then suitable for further testing, e.g. softening point testing in accordance with BS 2000-58 or penetration testing in accordance with BS 2000-49.

8.5 Reporting

Report the test results on the recovered material including a statement that dichloromethane has been used as the solvent. Give details of the test methods used on the recovered soluble binder.

NOTE The precision of the method has not been established.



NOTE Column may be Dufton-air, Dufton-vacuum jacketed or Vigreux.

CO_2 tube reaches to within 6 mm of the bottom; internal diameter of glass 4 mm to 6 mm.

Figure 6 — Distillation apparatus used for the recovery of soluble bitumen

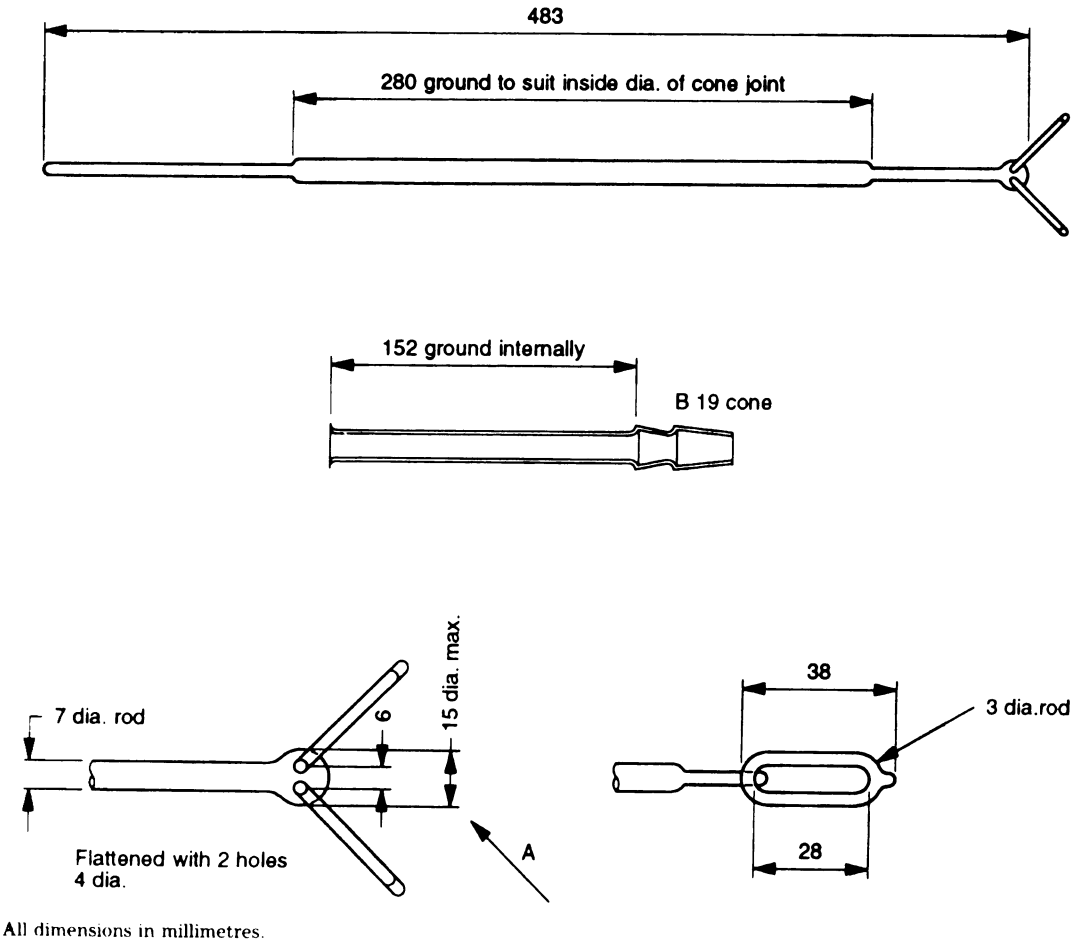


Figure 7 — Glass link stirrer

Annex A (informative) Safety

A.1 General

All possible aspects of safety cannot be covered in detail in this document. Every situation should be considered in its own right. Attention is drawn to the warning regarding the use of dichloromethane given in the foreword.

A.2 Protective clothing

Appropriate protective clothing should be worn whenever there is a risk to safety of the individual.

The following items should be considered:

a) safety helmets;

b) close-fitting overalls not coats;

c) goggles (see BS 2092);

d) face masks;

e) heat-resistant gloves or gauntlets;

f) safety boots;

g) reflective jackets.

A.3 Loose clothing

The sampler should not wear loose clothing, which might become entangled in machinery.

A.4 Safety principles

All personnel involved in sampling should receive instruction in the hazards associated with moving plant and vehicles, and be trained in the initial first aid treatment of burns to the skin due to contact with hot bituminous materials.

A.5 Safety equipment

All personnel should be aware of the position of and trained in the use of:

a) first aid kits;

b) fire extinguishers;

c) fire blankets;

d) mains power supply switches;

e) eye wash stations.

Annex B (informative) Recommendations for equipment control and calibration

B.1 General

Calibration of all critical equipment used in the performance of tests in this British Standard should be traceable to national standards as recommended in this annex. Systems used should conform to BS 5781-1.

B.2 External calibration

NOTE This is applicable to calibrations carried out under contract by an external organization.

Traceability should be established by the issue of a certificate for the relevant item giving details of the following:

a) the calibration;

b) the traceability route, i.e. British Calibration Service (BCS), National Physical Laboratory (NPL) or National Weights and Measures Laboratory (NWML);

c) the description and serial number of any reference/transfer standard involved;

d) the signature of the personnel performing or responsible for the calibration.

B.3 In-house calibration

NOTE This is applicable to calibration carried out by the staff of the testing laboratory.

When traceability is to be established by in-house calibration, the following conditions should be met:

a) the calibration should be performed in accordance with written procedures applicable to the apparatus;

b) the reference/transfer standards used should have valid certification provided by BCS, NPL, NWML or acceptable international bodies;

c) the reference/transfer standards should be used solely for calibration purposes;

d) the documentation should be as recommended in **B.2** and should be retained at least until the next calibration.

B.4 Balances and weights

B.4.1 Balances should be calibrated using reference weights at least once every 9 months; with frequent use 6 months is preferable.

B.4.2 Reference weights for in-house calibrations should be kept secure in a suitable environment, separate from working weights and should be recalibrated and certificated at least every 5 years.

B.5 Volumetric glassware

B.5.1 In-house calibration by weighing the amount of pure water that the vessel contains or delivers at a measured temperature is acceptable when used in conjunction with the corrections in BS 1797 and balances and weights that are in calibration and traceable.

B.5.2 Where the test method specifies class B glassware it is permissible to use uncalibrated class A glassware.

B.6 Centrifuges

B.6.1 It should be checked and recorded that the centrifuge is capable of producing the acceleration specified.

B.6.2 The centrifuge speed controls should be calibrated at the speed of rotation used in **B.6.1** at least every 6 months using a traceable tachometer or other method of similar accuracy.

B.6.3 The reference/transfer standard should be recalibrated and certificated at least once every 3 years.

B.7 Pressure gauges

B.7.1 Pressure gauges should be calibrated at least once every 6 months using a certificated reference gauge. Alternatively a mercury manometer corrected for local air pressure may be used as the reference standard.

B.7.2 The reference gauge should be recalibrated and certificated at least once every 2 years.

B.8 Stopclocks/watches

Stopclocks/watches should be calibrated at least once every 3 months.

B.9 Thermometry

B.9.1 For the methods described in this standard stamped, mercury-in-glass thermometers conforming to BS 593 are sufficient. Such thermometers should be recalibrated or replaced every 5 years as indicated in Appendix A of BS 593:1989.

B.9.2 If thermocouples are used they should be calibrated against a reference thermocouple, platinum resistance or reference mercury-in-glass thermometer at least once every 6 months.

B.9.3 Reference thermocouples or reference platinum resistance thermometers should be recalibrated at least once every 2 years and reference mercury-in-glass thermometers at least once every 5 years.

B.10 Test sieves

B.10.1 Only test sieves conforming to BS 410 should be used and their certificates retained throughout their working life.

B.10.2 Sieves should be marked with a set reference and only used as a constituent of that set. When new sieves are incorporated into a set they should be marked appropriately and the individual certificate filed.

B.10.3 Sieve control systems should include the following.

a) Visual checks should be made, by designated personnel, of the condition of all sieves at frequent intervals and the results recorded. The frequency of checks depends on volume of use but twice per week will be reasonable in most cases. Any discrepancies noted should be brought to the attention of a designated senior person who should decide whether to take action, i.e. recalibrate or replace the suspect sieve.

b) Standard gradings should be established from a laboratory standard sample of washed hard rock, and graded on each set of sieves in use at least once every 3 months. Alternatively gradings obtained using working sieves can be repeated on a set of sieves kept solely for calibration purposes and compared. The results of standard grading comparisons should be considered by designated personnel who should decide whether any individual sieve should be replaced or investigated further.

c) Measurement checks on the apertures of 300 mm diameter perforated-plate test sieves should be made in accordance with Appendix F of BS 410:1986 at least every 2 years of working use and more frequently if the sieves are in intensive use.

B.11 Bottle rotation machines

The speed of rotation of the bottles should be calibrated at least once per year.

B.12 Dichloromethane

Dichloromethane has to conform to BS 1994 at the time of purchase but once used and redistilled should be monitored as follows.

The residue on evaporation should be checked regularly by the boiling off procedure specified in sections 3 and 4 of BS 1994:1953, i.e. boil off duplicate aliquots of the recovered solvent and weigh the residue. Aliquots of 50 ml are recommended and the residue should not be greater than 0.005 g (i.e. 0.01 %). A frequency of once per week would be adequate for most testing laboratories.

List of references (see clause 2)

Normative references

BSI standards publications

BRITISH STANDARDS INSTITUTION, London

BS 410:1986, *Specification for test sieves.*
BS 812, *Testing aggregates.*
BS 812-103, *Method for determination of particle size distribution.*
BS 812-103.1:1985, *Sieve tests.*
BS 812-103.2:1989, *Sedimentation test.*
BS 1792:1982, *Specification for one-mark volumetric flasks.*
BS 1994:1953, *Specification for dichloromethane (methylene chloride).*
BS 4402:1982, *Specification for safety requirements for laboratory centrifuges.*

Informative references

BSI standards publications

BRITISH STANDARDS INSTITUTION, London

BS 593:1989, *Specification for laboratory thermometers.*
BS 598, *Sampling and examination of bituminous mixtures for roads and other paved areas.*
BS 598-100:1987, *Methods for sampling for analysis.*
BS 598-101:1987, *Methods for preparatory treatments of samples for analysis.*
BS 598-102:1989, *Analytical test methods.*
BS 1797:1987, *Schedule for tables for use in the calibration of volumetric glassware.*
BS 2000, *Methods of test for petroleum and its products.*
BS 2000-49:1983, *Penetration of bituminous materials.*
BS 2000-58:1988, *Softening point of bitumen (ring and ball).*
BS 2092:1987, *Specification for eye-protectors for industrial and non-industrial uses.*
BS 5781, *Quality assurance requirements for measuring equipment.*
BS 5781-1:1992, *Metrological confirmation system for measuring equipment.*

Other reference

[1] HSE Guidance Note EH/40 *Occupational exposure limits,* 1989[5)].

[5)] Available from HMSO, 49 High Holborn, London WC1 for personal callers or by post from HMSO, PO Box 276, London SW8 5DT.

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