
British Standard

Sampling and examination of bituminous mixtures for roads and other paved areas

Part 102. Analytical test methods

Echantillonnage et examen des mélanges bitumineux pour chaussées et autres surfaces pavées
Partie 102. Méthodes d'essais analytiques

Bituminöse Stoffe für Straßen und andere gedeckte Flächen; Probenahme und Prüfung
Teil 102. Analyseverfahren

Foreword

This Part of BS 598 has been prepared under the direction of the Road Engineering Standards Policy Committee. It is a revision of clauses 3 and 4 of BS 598 : Part 2 : 1974 which are now superseded by amendment. BS 598 : Part 1 : 1974 and BS 598 : Part 3 : 1985 are also being revised and as each of the tests or collection of related tests is reviewed the intention is to issue it as a separate Part or Section of this standard, the complete revision eventually comprising the following Parts:

- Part 100 Methods for sampling for analysis
- Part 101 Methods for preparatory treatment of samples for analysis
- Part 102 Analytical test methods
- *Part 103 Method for the recovery of soluble bitumen for examination
- *Part 104 Methods of test for the determination of density and compaction
- *Part 105 Methods of test for the determination of texture depth
- *Part 106 Methods of test for the determination of the stability index of pitch bitumen binders
- *Part 107 Methods of test for the determination of the target composition of wearing course rolled asphalt
- *Part 108 Methods of test for the determination of the condition of the binder on coated chippings and the rate of spread of coated chippings

It has been assumed in the drafting of this Part of BS 598 that the execution of its provisions is entrusted to appropriately qualified and experienced people.

This British Standard calls for the use of substances and/or procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage.

Appendix A describes the use of filter aids; appendix B gives recommendations for equipment control and calibration; appendix C lists the test sieves required for the methods of test contained in this standard; appendix D gives guidance on the various methods of test for determining binder content and grading of mineral aggregate and appendix E contains requirements for centrifuges.

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

WARNING. The methods described in this British Standard require the use of dichloromethane or trichloroethylene. These solvents are hazardous to health and are subject to occupational exposure limits as detailed in the latest edition of Guidance Note EH/40 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 these substances.

* In preparation.

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Methods

1 Scope

This Part of BS 598 describes analytical test methods to be carried out under laboratory test conditions on samples of bituminous mixtures.

The test methods described in this Part of BS 598 are not applicable to mastic asphalts; methods for testing these materials are described in BS 5284.

Test methods for modified binders are outside the scope of this standard because the methods require variations to the methods described in this standard.

NOTE. The titles of the publications referred to in this standard are listed on the inside back cover.

2 Definitions

For the purposes of this Part of BS 598 the definitions given in BS 6100 apply together with the following.

2.1 repeatability (r). The value below which the absolute difference between two single test results obtained with the same method on identical test material under the same conditions (same operator, same apparatus, same laboratory and a short interval of time) may be expected to lie with a specified probability; in the absence of other indications, the probability is 95 %.

2.2 reproducibility (R). The value below which the absolute difference between two single test results obtained with the same method on identical test material under different conditions (different operators, different apparatus, different laboratories and/or different time) may be expected to lie with a specified probability; in the absence of other indications, the probability is 95 %.

2.3 single test result. The value obtained by applying the standard test method fully, once, to a single specimen; it may be the mean of two or more observations or the result of a calculation from a set of observations as specified by the standardized test method.

2.4 test sieve. A sieve complying with the requirements of BS 410.

3 Limits for the repeatability and reproducibility of samples obtained from the same bulk sample

3.1 The limits for the differences between the analysis results of two test samples obtained from the same bulk sample shall be as given in table 1. The two test samples shall be obtained using the sampling procedure described in 3.2 or 3.3, as appropriate.

3.2 The test material for repeatability tests shall be obtained by dividing a sample twice the size required for a single test by the sample reduction procedure described in 3.5 of BS 598 : Part 101 : 1987.

3.3 The test material for reproducibility tests shall be obtained 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 using the sample reduction procedure described in 3.5 of BS 598 : Part 101 : 1987.

NOTE. Differences in the results obtained by two laboratories on test samples taken from the same bulk sample may be due to errors in the sample reduction and/or the analysis procedures adopted by the analysis. Any systematic difference in the comparison of a

Table 1. Repeatability and reproducibility limits for the analysis of coated macadams

Type of material	Nominal size larger than 20 mm		Nominal size 20 mm and smaller	
	Repeatability	Reproducibility	Repeatability	Reproducibility
Binder content	% 0.3	% 0.5	% 0.3	% 0.5
Grading aggregate passing BS test sieve				
37.5 mm	14	18	—	—
28 mm	12	15	—	—
20 mm	10	12	—	—
14 mm	10	12	7	9
10 mm	9	11	6	8
6.3 mm	8	10	5	7
3.35 mm	7	8	5	7
75 μ m				
Adjusted in accordance with 6.2.2	1.0	1.5	1.0	1.5

NOTE. The above figures are based on a repeatability and reproducibility exercise carried out several years ago on coated macadams. However, as the grading representation of BS 594 is now identical to that of BS 4987 the figures will also provide a reasonable guidance for hot rolled asphalt. It is possible that the precision will improve with the introduction of this standard and it is proposed to initiate a new exercise to investigate the position.

group of test results should be investigated to detect the cause or causes of the bias. 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 normally be possible, with co-operation between the laboratories, to eliminate the cause. If small differences occur due to the test methods being different, it is recommended that the interpretation of the test results, with respect to the specification, should be examined by the two laboratories concerned at the earliest opportunity.

4 Determination of binder content and grading of mineral aggregate

NOTE. For guidance see appendix D.

4.1 Extraction bottle method: Binder determination by difference

4.1.1 Apparatus

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

4.1.1.1 Metal bottles of capacity appropriate to the size of sample being analysed, e.g. 600 mL, 2.5 L, 7 L, 12 L with wide mouths and suitable closures.

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

4.1.1.2 A machine which will rotate the bottles about their longitudinal axes at a speed of 20 ± 10 r.p.m.

4.1.1.3 A pressure filter of appropriate size (one taking a filter paper of 270 mm diameter is suitable) and an air pump for supplying oil-free air at a pressure of at least 2 bar. If a filter aid is employed it shall be used in accordance with appendix A.

4.1.1.4 A supply of Whatman No 1 filter papers, or equivalent, to fit the pressure filter.

4.1.1.5 A funnel capable of supporting three 200 mm or 300 mm diameter sieves over the filling orifice of the pressure filter.

4.1.1.6 A set of test sieves complying with the requirements of BS 410, of appropriate diameters and aperture sizes (see appendix C).

4.1.1.7 A balance capable of weighing a sample to an accuracy of 0.05 % of its mass.

4.1.1.8 A sample divider, of size appropriate to the maximum particle size to be handled (see BS 812 : Part 102).

4.1.1.9 A number of trays that can be heated without damage or change in mass in which to dry recovered aggregate.

4.1.2 Solvent

4.1.2.1 Dichloromethane (methylene chloride), complying with the requirements of BS 1994 or *trichloroethylene* complying with BS 580.

NOTE. See **Warning** in the foreword.

4.1.3 Procedure

4.1.3.1 Obtain the sample by the methods specified in BS 598 : Parts 100 and 101. The mass of the sample shall be as given in table 2 of BS 598 : Part 101 : 1987.

4.1.3.2 If water is suspected in the sample, measure the water content by the method described in clause 5. It is essential that the water content is known as any errors in the estimation of water content will directly affect the estimate of binder content.

4.1.3.3 Weigh the sample to the nearest 0.05 % of the mass taken, and place it in a metal bottle (4.1.1.1) of appropriate size.

4.1.3.4 Add the solvent (4.1.2.1) to the sample to give a solution of not greater than 4 % concentration of soluble binder.

Caution. 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 or ejection of solvent on removal of the bung.

4.1.3.5 Close the bottle and roll it on the bottle rotating machine (4.1.1.2) for the time specified in table 2.

NOTE 1. For material which has been on the road for more than one month the extraction time may 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 exceed 30 % of the extraction time specified in table 2.

4.1.3.6 Weigh a dry filter paper (4.1.1.4) and fit it into the pressure filter (4.1.1.3). Support a nest of appropriate test sieves (4.1.1.6) above the funnel (4.1.1.5) mounted above the pressure filter. The finest test sieve shall be 75 µm size.

4.1.3.7 After the specified rolling time allow the contents to stand for at least 2 min so that filtration of the solution is made easier. Pour the solution through the test sieves into the pressure filter. Force the solution through the filter paper using air pressure.

4.1.3.8 Shake the aggregate remaining in the bottle with a further quantity of solvent, i.e. about 0.5 L. Immediately after shaking pour the solution through the nest of test sieves into the pressure filter, taking extreme care to ensure no loss of mineral matter. Then pass the solution through the pressure filter.

4.1.3.9 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 to remove as much of the mineral matter as possible and pass the final washings through the sieves and the pressure filter.

4.1.3.10 Transfer the aggregate on the sieves to a tray (4.1.1.9). Evaporate the solvent from the aggregate and the metal bottle. Transfer any mineral matter in the bottle to the tray with the remainder of the aggregate, taking care that all aggregate is removed from the bottle. Weigh and record the mass of the aggregate in the tray.

4.1.3.11 Remove the filter paper, complete with the mineral matter that passed the 75 µm test sieve, from the pressure filter. Dry and weigh; this will include the insoluble portion of the binder if insolubles were present in the binder. Deduct the mass of the filter paper.

Table 2. Time required for extraction (extraction bottle method: binder determination by difference)

Type of material	Minimum extraction time	
	tars	bitumens
Macadams other than dense, medium and fine graded macadams, containing cut-back bitumen or tar up to 44° e.v.t.*	min 30	min 5
Dense, medium and fine graded macadams containing cut-back bitumen or tar up to 44° e.v.t.	45	10
Macadams other than dense and medium graded macadams, containing penetration grade bitumen or tar over 44° e.v.t.	45	20
Rolled asphalt, dense tar surfacing, dense and medium graded macadams containing penetration grade bitumen or tar over 44° e.v.t.	60	30
Coated chippings	—	10

* e.v.t. = equiviscous temperature (see BS 76).

4.1.4 Calculations

4.1.4.1 Calculate the soluble binder content S (%) of the mass of the original dry sample by means of the following equation:

$$S = \frac{100(M - M_1) - MP}{M(100 - P)}$$

where

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

M_1 is the mass of aggregate and insoluble matter from the binder (in g);

P is the percentage by mass of water in the sample.

4.1.4.2 Where necessary, e.g. with tar binders, calculate the percentage by mass B of the total binder content on the dry sample:

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

where

T is the percentage by mass of binder insoluble in the solvent employed.

4.1.5 Grading of mineral aggregate

NOTE. Provided the aggregate was thoroughly washed, the proportion of material passing the 75 μ m 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 μ m test sieve found with the aggregate is greater than 1.5 % of the total aggregate, the result for the material passing the 75 μ m test sieve may be incorrect and is indicative of insufficient washing.

4.1.5.1 Determine the particle size distribution of all of the coarse-aggregate fraction on a nest of 300 mm diameter sieves in accordance with the requirements of BS 812: Part 103.

4.1.5.2 Determine the particle size distribution of the fine-aggregate fraction by dividing the fraction using a sample divider to obtain a sample of appropriate size complying with BS 812 : Part 103 with respect to the loading on each individual sieve. Determine the particle

size distribution of the fine aggregate on a nest of at least 200 mm diameter test sieves of appropriate mesh sizes in accordance with the requirements of BS 812 : Part 103. When the particle size distribution of only a portion of the fine-aggregate fraction is determined, multiply the masses of the graded fine aggregate by the ratio of the total mass of the fine-aggregate fraction to the mass of the portion taken.

NOTE. For each section of the grading the difference between the sum of the individual portions and the original weight should not exceed 0.5 % of the original weight.

4.1.5.3 Obtain the total mass of the material passing the 75 μ m test sieve by adding the mass of the material passing the 75 μ m test sieve from 4.1.5.2 to the mass of the material recovered from the pressure filter. If tar or other material containing insoluble binder was present in the sample the material passing the 75 μ m test sieve will include the insoluble portion of the binder, therefore calculate the amount of insoluble binder and subtract this from the amount passing the 75 μ m test sieve.

4.1.5.4 Calculate the grading of the total aggregate from 4.1.5.1 and 4.1.5.2.

4.1.6 *Reporting of results.* Report the results in accordance with clause 7 adjusted where appropriate in accordance with clause 6.

4.2 Extraction bottle method: Binder directly determined

4.2.1 Apparatus

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

4.2.1.1 *Metal bottles* of capacity appropriate to the size of sample being analysed, e.g. 600 mL, 2.5 L, 7 L, 12 L with wide mouths and suitable closures.

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

4.2.1.2 *A machine* which will rotate the bottles about their longitudinal axes at a speed of 20 $\pm$ 10 r.p.m.

4.2.1.3 Volumetric flasks of appropriate capacity, e.g. 250 mL, 500 mL, 1 L and 2 L complying with the requirements for grade B of BS 1792.

NOTE. Other systems of measuring the solvent may be employed provided that the apparatus used is calibrated in accordance with the requirements for grade B of BS 1792.

4.2.1.4 A centrifuge, complying with the requirements of appendix E.

4.2.1.5 A recovery apparatus comprising a water bath with an electric heater capable of maintaining boiling water in the bath throughout the recovery procedure, round-bottomed flasks of 200 mL or 250 mL capacity, a pressure gauge, a vacuum reservoir and a method of maintaining reduced pressure, e.g. a filter pump.

NOTE. Figure 1 illustrates a recovery apparatus but this particular arrangement is not mandatory provided that the conditions for recovering the binder from the solution as specified in 4.2.3.9, 4.2.3.10 and 4.2.3.11 are strictly followed.

4.2.1.6 A pressure filter of appropriate size (one taking a filter paper of 270 mm diameter is suitable) and an air pump for supplying oil-free air at a pressure of at least 2 bar. If a filter aid is employed it shall be used in accordance with appendix A.

4.2.1.7 A supply of Whatman No. 1 filter papers, or equivalent, to fit the pressure filter.

NOTE. Items 4.2.1.6 and 4.2.1.7 are not needed if the filler is to be determined by difference (see 4.2.5.2).

4.2.1.8 A set of test sieves, complying with the requirements of BS 410 of appropriate diameters and aperture sizes (see appendix C).

4.2.1.9 A burette of suitable size complying with the requirements for grade B of BS 846.

4.2.1.10 Balances of suitable capacities, capable of weighing samples to an accuracy of 0.05 % of their mass.

4.2.1.11 A balance accurate to 0.001 g for weighing flasks.

4.2.1.12 A stopclock or watch.

4.2.1.13 Containers resistant to solvent attack each with narrow neck and tight fitting resealable lid.

4.2.1.14 A sample divider of size appropriate to the maximum particle size to be handled.

4.2.1.15 Trays that can be heated without damage or change in mass, in which to dry recovered aggregate.

4.2.1.16 A desiccator to store the extraction flasks before weighing.

4.2.2 Solvent and reagent

4.2.2.1 Dichloromethane (methylene chloride), complying with the requirements of BS 1994.

NOTE. See **Warning** in the foreword.

4.2.2.2 Dried silica gel, crushed to pass a 75 µm test sieve.

NOTE. The silica gel should be dried to constant weight to 0.5 %.

4.2.3 Procedure

4.2.3.1 Obtain the sample by the methods specified in BS 598 : Parts 100 and 101. The mass of the sample shall be as given in table 2 of BS 598 : Part 101 : 1987. Allow the sample to cool.

4.2.3.2 Weigh the sample to the nearest 0.05 % of the mass taken, and place it in a metal bottle (4.2.1.1). To the same accuracy weigh sufficient silica gel (4.2.2.2) to absorb any water present and add to the bottle. The mass of silica gel shall be at least equal to the mass of water estimated to be present in the sample.

NOTE. Accurate weighing of the silica gel is not necessary if the fraction passing the 75 µm test sieve is to be determined by difference.

4.2.3.3 Add the required volume of dichloromethane (4.2.2.1) to the sample to give a solution of between 2 % and 4 % concentration of soluble binder. Measure the temperature of the solution.

NOTE 1. An estimate of the total volume V (in mL) of solvent required is given by the following formula:

$$V = \frac{M S_E}{C_S}$$

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 %).

The estimated volume V should be rounded to the nearest 250 mL.

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 4.2.3.3 and the temperature of the binder solution measured in accordance with 4.2.3.8 should not exceed ± 3 °C.

4.2.3.4 Close the bottle, and roll on the bottle rotating machine (4.2.1.2) for the time specified in table 3.

NOTE 1. For material which has been on the road for more than one month the extraction time may 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 exceed 30 % of the extraction time specified in table 3.

4.2.3.5 Remove the closed bottle from the rotating machine and stand it upright for a period of approximately 2 minutes to allow the bulk of the mineral matter to settle from suspension. Remove the stopper carefully from the closed bottle and, without delay, transfer approximately 500 mL of liquor to a clean dry pouring bottle. Transfer to the centrifuge tube(s) (4.2.1.4) sufficient liquor such that after centrifuging is complete there is enough solution to provide duplicate aliquot portions. Seal the remainder in the pouring bottle until aliquot portions are satisfactorily obtained. At all stages of this operation, keep the evaporation to a minimum by ensuring the containers (4.2.1.13) have close fitting lids and solutions are poured quickly. Seal the centrifuge tubes and centrifuge for the appropriate time determined from figure 2.

4.2.3.6 From this point carry out the remainder of the procedure in 4.2.3 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 4.2.3.8.

4.2.3.7 Dry a flask (4.2.1.3) and weigh the dry flask to the nearest 0.001 g.

Table 3. Time required for extraction (extraction bottle method: binder directly determined)

Type of material	Minimum extraction time	
	tars	bitumens
Macadams, other than dense, medium and fine graded macadams, containing cut-back bitumen or tar up to 44° e.v.t.*	min 30	min 5
Dense, medium and fine graded macadams containing cut-back bitumen or tar up to 44° e.v.t.	45	10
Macadams, other than dense and medium graded macadams, containing penetration grade binder or tar over 44° e.v.t.	45	20
Rolled asphalt, dense tar surfacing, dense and medium graded macadams containing penetration grade binder or tar over 44° e.v.t.	60	30
Coated chippings	—	10

* e.v.t. = equiviscous temperature (see BS 76).

4.2.3.8 Measure a sufficient amount of the centrifuged solution into the flask, using the burette (4.2.1.9), to give a residue of 0.75 g to 1.25 g of soluble binder after evaporation of the solvent.

Measure the temperature of the solution (see note 4 to 4.2.3.3).

NOTE. An estimate of the volume v (in mL) of solution (aliquot portion) required is given by the following formula:

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

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 v should be rounded to the nearest 5 mL.

4.2.3.9 Remove the solvent from the binder solution by connecting the flask to the recovery apparatus (4.2.1.5), 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 1. It is recommended that the distillation be carried out under reduced pressure. If reduced pressure is used the pressure should be not less than 600 mbar.

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.

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

(a) For penetration grade bitumen and tars of 44° e.v.t. and higher reduce the pressure to between 180 mbar and 220 mbar in 1 min to 2 min and maintain at this pressure for a further 3 min to 4 min.

(b) For cut-back bitumens, and tars below 44° e.v.t. allow the pressure to increase to approximately atmospheric pressure and then reduce it to between 550 mbar and 650 mbar in 1 min to 2 min and maintain it at this pressure for a further 3 min to 4 min.

4.2.3.11 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 any 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.

4.2.3.12 Cool the flask in a desiccator (4.2.1.16) and weigh to the nearest 0.001 g. If the mass of soluble binder recovered is not between 0.75 g and 1.25 g (inclusive) repeat the procedure from 4.2.3.6 to 4.2.3.12 inclusive with another portion of the solution after adjusting the volume of the aliquot (see 4.2.3.8) to ensure that an adequate mass of binder is recovered.

If the difference between the duplicate recoveries is greater than 0.02 g 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.

4.2.4 Calculations

4.2.4.1 Calculate the soluble binder content S (%) of the mass of the original dry sample by means of the following equation:

$$S = \frac{10\,000 z V}{v M (100 - P) (1 - z/dv)}$$

where

- M is the mass of undried sample (in g);
- z is the average mass of binder recovered from each of two aliquot portions (in g);
- V is the total volume of solvent (in mL);
- v is the volume of each aliquot portion (in mL) (see 4.2.3.8);
- d is the relative density, 15.5/15.5 °C of the binder;
- P is the percentage by mass of water in the sample (see clause 5).

4.2.4.2 Where necessary, e.g. with tar binders, calculate the percentage by mass B of the total binder content on the dry sample by means of the following equation:

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

where

- T is the percentage by mass of binder insoluble in the solvent employed.

4.2.5 Washing of mineral aggregate

4.2.5.1 Filler, directly determined

4.2.5.1.1 After removing sufficient solution for the determination of the soluble binder content pour the liquid contents of the extraction bottle (including fine matter in suspension, but taking care not to carry over any other aggregate) through a 75 µm test sieve (4.2.1.8) protected by a 1.18 mm test sieve and through the funnel into the pressure filter (4.2.1.6).

4.2.5.1.2 Fit the pressure filter with a Whatman no. 1 filter paper or equivalent (4.2.1.7).

4.2.5.1.3 Pass the liquid through the filter paper under air pressure of at least 2 bar.

4.2.5.1.4 Shake the aggregate remaining in the bottle with a further quantity of solvent, i.e. about half the quantity of solvent used originally (see 4.2.3.3) or about 1 L, whichever is the smaller. Immediately after shaking pour the solution through the nest of sieves into the pressure filter, ensuring no loss of mineral matter. Pass the solution through the pressure filter. Repeat 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 to a suitably sized tray (4.2.1.15). Rinse the bottle once more to remove as much of the mineral matter as possible and pour the final washings through the 75 µm test sieve and pass them through the pressure filter. Any mineral matter in the portion of solution removed for the determination of binder content shall be recovered with the rest of the aggregate.

NOTE. A filter aid as described in appendix A may be used to speed up filtration.

4.2.5.2 Filler determined by difference

4.2.5.2.1 After removing sufficient solution for the determination of the soluble binder content pour the liquid contents of the extraction bottle (including fine matter in suspension, minimizing as far as possible the carrying over of any other aggregate) through a 75 µm test sieve (4.2.1.8) protected by a 1.18 mm test sieve to waste.

4.2.5.2.2 Shake the aggregate remaining in the bottle with a further quantity of solvent, i.e. about half the quantity of solvent used originally (see 4.2.3.3) or about 1 L, whichever is the smaller. Immediately after shaking pour the solution through the nest of sieves, ensuring no loss of mineral matter. 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 bulk of the contents of the bottle to a suitably sized tray (4.2.1.15). Rinse the bottle once more to remove as much of the mineral matter as possible and pour the final washings through the 75 µm test sieve.

4.2.6 Grading of mineral aggregate

NOTE. 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.

4.2.6.1 Evaporate the solvent from and combine the aggregate on the sieves, on the tray and in the bottle.

4.2.6.2 Determine the particle size distribution of all of the coarse-aggregate fraction on a nest of 300 mm diameter sieves appropriate for the mixture in accordance with the requirements of BS 812 : Part 103.

4.2.6.3 Determine the particle size distribution of the fine-aggregate fraction by taking a sample of 100 g to 200 g. Where required divide the fine-aggregate fraction, using a sample divider. Determine the particle size distribution of the fine aggregate on a nest of 200 mm diameter test sieves of appropriate mesh sizes in accordance with the requirements of BS 812 : Part 103. When the particle size distribution of only a portion of the fine-aggregate fraction is determined multiply the masses of the graded fine aggregate by the ratio of the total mass of the fine-aggregate fraction to the mass of the portion taken.

4.2.6.4 If the filler content is being directly determined, dry and weigh the material passing the 75 µm test sieve recovered from the pressure filter. To obtain the total mass of the material passing the 75 µm test sieve, add the mass of the material passing the 75 µm test sieve from 4.2.6.3 to the mass of the material recovered from the pressure filter.

NOTE. The total mass will include the silica gel added before extraction together with the insoluble portion of the binder.

4.2.6.5 Calculate the grading of the total aggregate from 4.2.6.2 and 4.2.6.3.

4.2.6.6 Alternatively, the mass M_F (in g) of the aggregate passing the 75 µm test sieve is calculated by difference as follows after making allowance for any water present in the sample:

$$M_F = M_A - M_W - M_B - M_C$$

where

M_A is the total mass of aggregate (in g)

M_W is the mass of water (in g)

M_B is the mass of total binder (in g)

M_C is the mass of material retained on the 75 µm test sieve (in g).