

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

Part 102: Analytical test methods

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- County Surveyors' Society
- Department of the Environment
- Department of Transport (Highways Agency)
- Federation of Civil Engineering Contractors
- Institution of Civil Engineers
- Local Authority Organizations
- Road Surface Dressing Association
- Sand and Gravel Association Limited
- Society of Chemical Industry

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- British Civil Engineering Test Equipment Manufacturers' Association
- Department of Transport (Transport Research Laboratory)
- Institute of Asphalt Technology
- Institute of Petroleum
- Institution of Highways and Transportation
- Mastic Asphalt Producers' Association
- Refined Bitumen Association Ltd.
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Foreword

This part of BS 598 has been prepared by Subcommittee B/510/1. It supersedes BS 598-102:1989 which is withdrawn.

This part of BS 598 introduces technical changes to the methods for determining binder content and grading of mineral aggregate, the use of filter aids and to recommendations for equipment control and calibration. This edition does not reflect a full revision of the standard because of parallel work proceeding in CEN. 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.

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.

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 precision and it is emphasized that adequate training should be given to staff employed in the usage of these substances.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 24, an inside back cover and a back cover.

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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 bituminous materials made with modified binders are outside the scope of this standard. Experience has shown that use of the methods described in this standard will only provide an approximate binder content for such mixtures.

NOTE It may be possible to obtain accurate binder content results by use of an adjustment factor for insoluble matter based on information provided by the modified binder supplier.

2 References

2.1 Normative references

This part of BS 598 incorporates, by dated or undated reference, provisions from other publications. These normative references are made at the appropriate places in the text and the cited publications are listed on the inside back cover. For dated references, only the edition cited applies; any subsequent amendments to or revisions of the cited publication apply to this part of BS 598 only when incorporated in the reference by amendment or revision. For undated references, the latest edition of the cited publication applies, together with any amendments.

2.2 Informative references

This part of BS 598 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 the latest editions.

3 Definitions

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

3.1 repeatability (*r*)

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 within a specified probability; in the absence of other indications, the probability is 95 %

3.2 reproducibility (*R*)

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 within a specified probability; in the absence of other indications, the probability is 95 %

3.3 single test result

value obtained by applying the 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 test method

3.4 test sieve

sieve conforming to BS 410

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

4.1 The limits for the difference 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 4.2 or 4.3, as appropriate.

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

4.3 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 598-101:1987.

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 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 cooperation 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 should be examined by the two laboratories concerned at the earliest opportunity.

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 µm Adjusted in accordance with 7.2.2	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 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.				

5 Determination of binder content and grading of mineral aggregate

NOTE For guidance see annex D.

5.1 Extraction bottle method: binder determination by difference

5.1.1 Apparatus

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

5.1.1.1 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 filled to more than three-quarters full.

5.1.1.2 Machine, which will rotate the bottles about their longitudinal axes at a speed of (20 ± 10) r/min.

5.1.1.3 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 bar¹⁾. If a filter aid is employed it shall be used in accordance with annex A.

5.1.1.4 Supply of Whatman no. 1 filter papers, or equivalent, to fit the pressure filter.

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

5.1.1.6 Set of test sieves, conforming to BS 410, of appropriate diameters and aperture sizes (see annex C).

5.1.1.7 Balance, capable of weighing a sample to an accuracy of 0.05 % of its mass.

5.1.1.8 Sample divider, of size appropriate to the maximum particle size to be handled (see BS 812-102).

5.1.1.9 Number of trays, that can be heated without damage or change in mass in which to dry recovered aggregate.

5.1.2 Solvent

5.1.2.1 Dichloromethane (methylene chloride), conforming to the requirements of BS 1994.

5.1.2.2 Trichloroethylene, which shall be clear, almost colourless and free from matter in suspension. It shall have a minimum assay of 95 %.

NOTE 1 See **WARNING** in the foreword.

NOTE 2 When trichloroethylene is recovered by distillation for further use, acidity may develop. A useful precaution is to store the solvent over calcium oxide in coloured glass or a suitable metal container.

5.1.3 Procedure

5.1.3.1 Obtain the sample by the methods specified in BS 598-100 and 598-101. The mass of the sample shall be as given in Table 2 of BS 598-101:1987.

¹⁾ 1 bar = 100 kN/m² = 100 kPa.

5.1.3.2 If water is suspected to be present in the sample, measure the water content by the method described in clause 6. 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.

5.1.3.3 Weigh the sample to the nearest 0.05 % of the mass taken, and place it in a metal bottle (5.1.1.1) of appropriate size.

5.1.3.4 Add the solvent (5.1.2.1 or 5.1.2.2) 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.

5.1.3.5 Close the bottle and roll it on the bottle rotating machine (5.1.1.2) for the time given 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 given in Table 2.

**Table 2 — Time required for extraction
(extraction bottle method: binder
determination by difference) —
Minimum extraction time**

Type of material	Tars min	Bitumens min
Macadams other than dense, close, medium or fine graded. Macadams containing cut-back bitumens	30	10
Rolled asphalt, dense tar surfacing, dense, close, medium and fine graded macadams containing penetration grade bitumen	60	20
Coated chippings	—	10

5.1.3.6 Weigh a dry filter paper (5.1.1.4) and fit it into the pressure filter (5.1.1.3). Support a nest of appropriate test sieves (5.1.1.6) above the funnel (5.1.1.5) mounted above the pressure filter. The finest test sieve shall be 75 µm size.

5.1.3.7 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.

5.1.3.8 Shake the aggregate remaining in the bottle with a further quantity of solvent, i.e. about half the quantity of solvent used originally (see 5.1.3.4) 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 solvent 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 (5.1.1.9). Rinse the bottle once more to remove as much of the mineral matter as possible, 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 annex A may be used to speed up filtration.

5.1.3.9 Transfer the aggregate on the sieves to a tray (5.1.1.9). 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.

5.1.3.10 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.

5.1.4 Calculations

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

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

where

- M is the mass of undried sample (in grams);
- M_1 is the mass of aggregate and insoluble matter from the binder (in grams);
- P is the percentage by mass of water in the sample.

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

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

where

- S is the soluble binder content as a percentage;
 T is the percentage by mass of binder insoluble in the solvent employed.

5.1.5 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 found 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.

5.1.5.1 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 812-103.

5.1.5.2 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 conforming to BS 812-103 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 812-103. 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 2 For each fraction of the grading the difference between the sum of the individual portions and the original mass should not exceed 0.5 % of the original mass.

5.1.5.3 Obtain the total mass of the material passing the 75 µm test sieve by adding the mass of the material passing the 75 µm test sieve from **5.1.5.2** 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 µm 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 µm test sieve.

5.1.5.4 Calculate the grading of the total aggregate from **5.1.5.1** and **5.1.5.2**.

5.1.5.5 Report the results in accordance with clause 8 adjusted where appropriate in accordance with clause 7.

5.2 Extraction bottle method: binder directly determined

5.2.1 Apparatus

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

5.2.1.1 *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.

5.2.1.2 *Machine*, which will rotate the bottles about their longitudinal axes at a speed of (20 ± 10) r/min.

5.2.1.3 *Volumetric flasks* of appropriate capacity, e.g. 250 ml, 500 ml, 1 l and 2 l conforming to 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.

5.2.1.4 *Centrifuge*, see annex E.

5.2.1.5 *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 vacuum pump.

NOTE Figure 1 illustrates a recovery apparatus but this particular arrangement is not a requirement provided that the conditions for recovering the binder from the solution as specified in **5.2.3.9**, **5.2.3.10** and **5.2.3.11** are followed.

5.2.1.6 *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 annex A.

5.2.1.7 *Supply of Whatman no.1 filter papers*, or equivalent, to fit the pressure filter.

NOTE Items **5.2.1.6** and **5.2.1.7** are not needed if the filler is to be determined by difference (see **5.2.5.2**).

5.2.1.8 *Set of test sieves*, conforming to BS 410 of appropriate diameters and aperture sizes (see annex C).

5.2.1.9 *Burette*, of suitable size conforming to the requirements for grade B of BS 846.

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

5.2.1.11 *Balance*, accurate to 0.001 g for weighing flasks.

5.2.1.12 *Stopclock or watch*.

5.2.1.13 *Containers*, resistant to solvent attack each with narrow neck and tight fitting resealable lid.

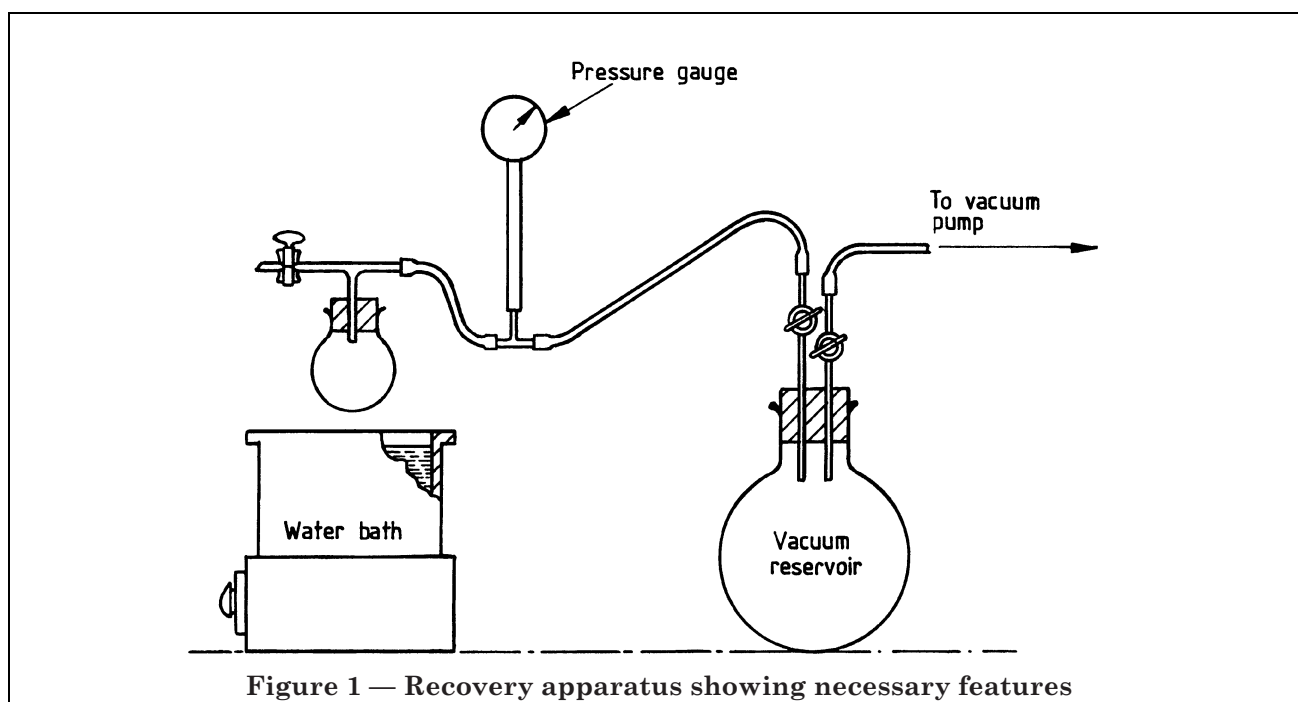


Figure 1 — Recovery apparatus showing necessary features

5.2.1.14 *Sample divider*, of size appropriate to the maximum particle size to be handled.

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

5.2.1.16 *Desiccator*, to store the extraction flasks before weighing.

5.2.2 Solvent and reagent

5.2.2.1 *Dichloromethane (methylene chloride)*, conforming to BS 1994.

NOTE See **WARNING** in the foreword.

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

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

5.2.3 Procedure

5.2.3.1 Obtain the sample by the methods specified in BS 598-100 and BS 598-101. The mass of the sample shall be as given in Table 2 of BS 598-101:1987. Allow the sample to cool.

5.2.3.2 Weigh the sample to the nearest 0.05 % of the mass taken, and place it in a metal bottle (**5.2.1.1**). To the same accuracy weigh sufficient silica gel (**5.2.2.2**) 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.

5.2.3.3 Measure and record the temperature of the dichloromethane immediately prior to adding the required volume to the sample. The volume added shall give a solution of between 2 % and 4 % concentration of soluble binder.

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

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

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 %).

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 **5.2.3.3** and the temperature of the binder solution when measured in accordance with **5.2.3.8** should not exceed ± 3 °C. Temperature measurement is best conducted using a rapid response electronic thermometer with a low thermal capacity probe in order to minimize evaporational loss during the process.

NOTE 5 Where the temperature of the binder solution (**5.2.3.8**) is outside the range ± 3 °C of the temperature of the dichloromethane (**5.2.3.3**) gentle heating or cooling of the solution is permitted providing evaporation of the solvent is prevented.