



Testing aggregates —

Part 119: Method for determination of acid-soluble material in fine aggregate

UDC [625.07 + 691.22]:[543.726 + 543.832]

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Cement, Gypsum, Aggregates and Quarry Products Standards Committee (CAB/-) to Technical Committee CAB/2 upon which the following bodies were represented:

Aggregate Concrete Block Association
 Association of Consulting Engineers
 Association of Consulting Scientists
 Association of Lightweight Aggregate Manufacturers
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 Sand and Gravel Association Limited
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This British Standard, having been prepared under the direction of the Cement, Gypsum, Aggregates and Quarry Products Standards Committee, was published under the authority of the Board of BSI and comes into effect on 31 July 1985

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The following BSI references relate to the work on this standard:
 Committee reference CAB/2
 Draft for comment 82/14681 DC

ISBN 0 580 14544 1

Amendments issued since publication

Amd. No.	Date of issue	Comments

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Foreword

This Part of BS 812, prepared under the direction of the Cement, Gypsum, Aggregates and Quarry Products Standards Committee, has been produced for the particular purpose of testing fine aggregate to be used in concrete road surfaces and concrete paving blocks. BS 812-2, BS 812-3 and BS 812-4 are also being revised and as each of the tests, or collections of related tests, is revised, it is intended to issue it as a separate Part or Section of this standard.

It is intended that other British Standards should call up BS 812 test methods as the basis of compliance. Nevertheless, it is *not* intended that all aggregates should be subjected regularly to all the listed tests. Specifications in other standards will refer only to the relevant test methods.

Some of the tests in other Parts of this standard are of limited application, and advice on the use of simpler tests is given, for example, when they can be used for a preliminary sorting of aggregates to see whether more expensive testing is justified.

It is necessary that pavement surfaces do not develop a polished surface of low skidding resistance when trafficked and it is desirable that pavement surfaces be tested for resistance to polishing. However, such a test has not yet been published as a British Standard and some authorities exclude the use of fine aggregates containing significant amounts of acid-soluble material, some of which have been found to give poor polishing resistance. Such aggregates are excluded only because of their low resistance to polishing although they have no adverse effects on any of the other properties of the concrete.

The procedure for dividing the test sample into two fractions, one passing a 5.0 mm test sieve and retained on a 600 μm test sieve and the other passing a 600 μm test sieve, and making separate determinations of acid-soluble material on each fraction fulfils the special needs of the authorities concerned with specifying fine aggregate to be used in concrete pavement surfaces.

The procedure for the determination of acid-soluble material (clause 8) may have other applications but it should be realized that the results so obtained may not correlate with processes involving, for example, other acids or acid concentrations.

Reference should be made to BS 812-101 for general guidance on testing aggregates, precision of test methods and variance arising from sampling errors. 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 4, 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 Part of BS 812 describes a method of determining the amount of acid-soluble material in both of the size fractions of fine aggregate, all passing a 5.00 mm test sieve, separated by sieving on a 600 μm test sieve.

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 812 the definitions given in BS 812-101 and BS 812-102 apply.

3 Principle

The fraction of fine aggregate passing a 5.00 mm test sieve is separated by sieving on a 600 μm test sieve. A weighed test portion of each fraction is treated with hydrochloric acid and the dried residue weighed, when cool, to determine the mass of acid-soluble material extracted.

NOTE The original intention of the test was to measure acid-soluble carbonates. With some materials, however, other acid-soluble components may be present and these may contribute to a high value, particularly if digestion with acid is prolonged.

4 Sampling

The sample used for the test (the laboratory sample) shall be taken in accordance with the procedure described in clause 5 of BS 812-102:1984.

5 Apparatus

5.1 A *sample divider*, of size appropriate to use with fine aggregate, e.g. a riffle box as specified in BS 812-102 or, alternatively, a flat scoop and a clean, flat and hard horizontal surface, e.g. a metal tray, for use in quartering.

5.2 *Test sieves*, with apertures of 5.00 mm and 600 μm , complying with BS 410 and with fitting lids and receivers.

5.3 A *balance*, of 100 g capacity, accurate to 0.1 g or less.

5.4 *Two beakers* or *two wide-mouth conical flasks*, of 500 mL capacity.

5.5 *Medium grade filter papers*¹⁾.

5.6 *Two filter funnels*, appropriate to the diameter of the filter papers.

5.7 A *measuring cylinder*, of 25 mL capacity.

5.8 An *electric hot plate*, capable of being controlled to maintain the temperature of the contents of the flasks at just below boiling point.

5.9 A *wash bottle*.

5.10 *Two evaporating basins*, of about 250 mL capacity.

5.11 A *drying oven*, capable of being controlled to maintain a temperature of 105 ± 5 °C.

5.12 A *desiccator*, of sufficient size to hold the evaporating basin.

6 Reagents

6.1 *Hydrochloric acid solution* (approximately 4 mol/L), prepared by diluting 360 mL of concentrated hydrochloric acid (p1.18) to 1 L with distilled water.

6.2 *Distilled water*, complying with BS 3978 or, alternatively, *deionized water*.

7 Preparation of test portion

7.1 Test sample

Reduce the laboratory sample by the procedures described in clause 6 of BS 812-102:1984 to produce a sub-sample of mass approximately 400 g. If sub-division is by using a riffle box, ensure that the sample is free flowing by drying it if necessary. Sieve the sub-sample through a 5.00 mm test sieve rejecting any particles retained on the sieve. The material passing the sieve is the test sample.

7.2 Test portion

7.2.1 Dry the test sample by heating at a temperature of 105 ± 5 °C until constant mass is achieved, cool and sieve through a 600 μm test sieve and collect the fraction passing the sieve in a fitting receiver. To prevent blinding of the sieve apertures by overloading, place an amount of aggregate on the sieve such that the amount retained on completion of sieving is not greater than 75 g on a 200 mm diameter sieve or not greater than 110 g on a 300 mm diameter sieve. Continue sieving until not more than 1 g of undersize material passes the sieve during 1 min. If mechanical sieving is used, check that separation is complete by hand sieving briefly.

7.2.2 If either the material retained on the sieve or that passing has a mass of less than 50 g, repeat the procedure described in **7.2.1** on a new test sample produced by the procedure described in **7.1** to produce a total of at least 50 g oversize and 50 g undersize fractions.

¹⁾ Whatman No. 40 filter papers have been found to be suitable.

NOTE When sieve analysis using the procedures described in BS 812-103²⁾ has been carried out it is permissible to obtain the two fractions required by combining the material passing a 5.00 mm test sieve and retained on all the test sieves with apertures of 600 µm or larger and all the material passing the 600 µm test sieve.

7.2.3 Use the procedures described in clause 6 of BS 812-102:1984 to reduce the material retained on the 600 µm test sieve to provide a test portion with a mass of 50 ± 5 g. Weigh the test portion and record its mass to the nearest 0.1 g (M_1).

7.2.4 Similarly reduce the material passing the 600 µm test sieve to produce a weighed test portion with a mass of 50 ± 5 g.

NOTE For some applications the procedure of separating the test sample on a 600 µm test sieve is not required. In those cases the dried test sample should be reduced to produce a weighed test portion with a mass of 50 ± 5 g.

8 Procedure

8.1 General

Determine, separately, the acid-soluble material content of both test portions (see 7.2) by the method described in 8.2.

NOTE Material which contains clay (see 8.2.4) may be difficult to filter and may give anomalous results. Improvement of filtration may be achieved by adding 1 mL of polyacrylamide solution (1 g polyacrylamide of relative molecular mass $< 5 \times 10^6$, dissolved in 1 L of distilled water) to the suspension to flocculate suspended matter.

8.2 Test procedure

8.2.1 Dry a medium grade filter paper (5.5) by heating in an oven (5.11) at a temperature of 105 ± 5 °C for 60 ± 15 min. Cool at room temperature for 30 min, weigh and record its mass to the nearest 0.1 g (M_2).

8.2.2 Transfer the weighed test portion of fine aggregate fraction to a 500 mL beaker or conical flask (5.4) and add 25 mL of the dilute hydrochloric acid solution (6.1) and agitate the contents of the flask. When effervescence is considerable add the acid with care to avoid loss of material. When any effervescence has ceased add a further 25 mL of dilute acid and agitate. Continue adding 25 mL portions of the dilute hydrochloric acid until no further effervescence occurs on adding the acid.

8.2.3 Heat the flask on the electric hot plate (5.8) until its contents are hot but not boiling, i.e. 70 °C to 90 °C, and maintain at this temperature for 5 min to 10 min. Add a further 25 mL of dilute hydrochloric acid to the hot solution to confirm that reaction has ceased. If further effervescence occurs, continue adding 25 mL portions of the dilute hydrochloric acid to the hot solution until no further effervescence occurs on adding acid.

NOTE No further effervescence from the aggregate particles is taken as an indication of cessation of reaction. Reaction with acid should not be continued for more than approximately 1 h.

8.2.4 Remove the flask from the source of heat and decant the solution through the weighed filter paper supported in a suitable filter funnel (5.6). Add 50 mL hot, but not boiling, distilled water (5.2) to the residual undissolved aggregate in the flask, agitate and decant the water through the filter paper. Repeat the washing and decanting process at least five times.

NOTE Vacuum filtration may give an increase in filtration rate.

8.2.5 Wash the residue from the flask into an evaporating basin (5.10) (see note) with water, ensuring that all the undissolved aggregate is transferred. Decant the supernatant liquid from the basin through the filter paper. When all the liquid has been drained, place the filter paper and its contents in the evaporating basin with the residue and dry in an oven at a temperature of 105 ± 5 °C for 16 ± 1 h or until constant mass is achieved. Cool to room temperature in a desiccator (5.12). Weigh the dried residual aggregate plus the filter paper and its contents and record the mass to the nearest 0.1 g (M_3).

NOTE The evaporating basin may be pre-weighed after drying and cooling. In this case the dried residual aggregate is weighed together with the basin and the filter paper.

9 Calculation and expressions of results

Calculate the percentage loss in mass caused by acid treatment from the equation:

$$\% \text{ acid-soluble material} = \frac{M_1 + M_2 - M_3}{M_1} \times 100$$

where

M_1 is the mass of dried test portion (in g);

M_2 is the mass of dried filter paper (in g);

M_3 is the mass of dried residue plus filter paper (in g).

NOTE If a pre-weighed evaporating basin has been used the values of M_2 and M_3 are slightly modified as follows:

M_2 is the mass of dried filter paper plus evaporating basin (in g);

M_3 is the mass of dried residue plus filter paper plus evaporating basin (in g).

Express the result as the percentage acid-soluble material content of the sample to the nearest 1 %.

²⁾ In preparation.

10 Precision

The precision of the test method, including the taking of the laboratory samples according to the procedure described in clause 5 of BS 812-102:1984 for sampling from heaps, has been measured for two types of material. The results are given in Table 1.

11 Test report

The test report shall affirm that the acid-soluble material content was determined in accordance with this Part of BS 812, and whether or not a certificate of sampling is available. If available, a copy of the certificate of sampling shall be provided. The test report shall include the following additional information:

- a) sample identification;
- b) either:
 - 1) the acid-soluble material content of each of the fractions of fine aggregate portion retained and passing a 600 μm test sieve; or
 - 2) the acid-soluble material content of the fine aggregate portion.

Table 1 — Examples of precision data for determination of the content of acid-soluble material

Type of aggregate	Size fraction	\bar{x}	r_1	R_1	R_2	$\sqrt{V_{r1}}$	$\sqrt{V_L}$	$\sqrt{V_S}$
Sand with shell particles ^a	5.0 mm to 600 μm	%	%	%	%	%	%	%
	Passing 600 μm	59.0	3.3	5.0	5.1	1.20	1.31	0.45
Sand with limestone and other acid soluble particles ^b	5.0 mm to 600 μm	19.5	0.7	1.2	1.3	0.24	0.37	0.10
	Passing 600 μm	16.7	4.9	6.7	9.4	1.76	1.62	2.34
		11.2	2.6	5.8	6.4	0.94	1.86	0.90

^a The precision data for this material were determined from an experiment conducted in 1982 involving 48 laboratory samples, taken from a 10 tonne lot of the material, and 24 laboratories. The tests for outliers given in BS 5497-1:1979 were applied to the data. All the test results from one laboratory obtained on the passing 600 μm fractions were rejected because their average was an outlier. The test results obtained on the passing 600 μm fraction of four laboratory samples, and those obtained on the 600 μm to 5.0 mm fraction of one laboratory sample, were rejected because for each of these laboratory samples the difference between the two test results on the laboratory sample was an outlier.

^b The precision data for this material were determined from an experiment conducted in 1984 involving 16 laboratory samples, taken from a 20 tonne lot of the material, and 8 laboratories. The tests for outliers given in BS 5497-1:1979 were applied to the data. All the test results from one laboratory obtained on the 5.0 mm to 600 μm fraction and on the passing 600 μm fraction were rejected because their averages were outliers.

NOTE 1 The table quotes values for the square roots of V_{r1} , V_L and V_S so that these values have the same units of measurement as the other values in the table.

NOTE 2 Definitions of \bar{x} , r_1 , R_1 , R_2 , V_{r1} , V_L and V_S and the uses of precision data are given in BS 812-101.

Publications referred to

BS 410, *Specification for test sieves*.

BS 812, *Testing aggregates*.

BS 812-2, *Methods for determination of physical properties*³⁾.

BS 812-3, *Methods for determination of mechanical properties*³⁾.

BS 812-4, *Methods for determination of chemical properties*³⁾.

BS 812-101, *Guide to sampling and testing aggregates*.

BS 812-102, *Methods for sampling*.

BS 812-103, *Methods for determination of particle size distribution*⁴⁾.

BS 3978, *Water for laboratory use*.

BS 5497, *Precision of test methods*.

BS 5497-1, *Guide for the determination of repeatability and reproducibility for a standard test method*.

³⁾ Referred to in the foreword only.

⁴⁾ In preparation.

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