

Methods of testing mortars, screeds and plasters

Part 2. Chemical analysis and aggregate grading

ICS 91.100.10

Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Technical Committee B/519, Masonry and associated testing, upon which the following bodies were represented:

- Autoclaved Aerated Concrete Products Association
- Brick Development Association
- British Ceramic Research Ltd.
- Building Employers' Confederation
- Concrete Block Association
- Department of the Environment (Building Research Establishment)
- District Surveyors' Association
- Local Authority Organizations
- Mortar Producers Association Limited
- National House Building Council
- Royal Institute of British Architects

The following bodies were also represented in the drafting of the standard, through a subcommittee:

- British Aggregate Construction Materials Industries
- British Cement Association
- British Civil Engineering Test Equipment Manufacturers' Association
- British Lime Association
- British Vermiculite Association
- Cement Admixtures Association
- Cementitious Slag Makers' Association
- Electricity Association
- Institute of Concrete Technology
- Quality Ash Association

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Foreword

This British Standard has been prepared under the direction of Technical Committee B/519, and is a revision of section 1 and section 2 of BS 4551 : 1980. The main purpose of this revision has been to introduce precision data for the individual methods described. BS 4551 : Part 2 : 1998, together with Part 1 : 1998, supersedes BS 4551 : 1980, which is withdrawn.

It is impossible to relate the analytical results to volume proportions of a mortar mix with any great accuracy. A dry mass basis has been introduced, which it is recommended should eventually replace the use of volume proportions.

The precision data in this Part of BS 4551 were obtained using the classical methods described. Instrumental methods of analysis are permitted, but are not defined because there is, as yet, no general agreement on the details of appropriate methods. The committee responsible for this revision welcomes comments on details of proposed instrumental methods of analysis, particularly for soluble silica, and will incorporate such methods if persuaded of their validity. As far as is possible, the methods described in this Part of BS 4551 are the same as those in BS 1881 : Part 124 *Methods for analysis of hardened concrete*.

Attention is drawn to the wide range of cements which will be available following the publication of new European Standards. This may cause considerable problems of interpretation when the ingredients of the mortars being analysed are not known with any certainty.

Although it is desirable that tests suitable for site use are included in this standard, insufficient evidence is available to date for these to be included in the present revision. The testing of bricks or blocks or stone and mortars and plasters together is considered to be the most satisfactory approach for determining the quality of masonry, and in BS 5628 : Part 1 this principle is adopted.

This Part of BS 4551 calls for the use of substances and procedures that may be injurious to health if adequate precautions are not taken. It refers only to their technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage.

Compliance with a British Standard does not 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 24, an inside back cover and a back cover.



Method

1 Scope

This British Standard specifies methods of sampling, laboratory preparation, chemical analysis and aggregate grading of mortars for bricklaying, screeding, plastering and rendering. Methods of interpretation of chemical analysis results are also specified.

The methods given in this standard are not intended to be applied to mortars containing high alumina cement.

2 References

2.1 Normative references

This Part of BS 4551 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 4551 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 4551 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 British Standard the definitions given in BS 6100 apply.

4 Sampling

4.1 Principle

This clause deals with the sampling of mortars and their subsequent reduction on site to a quantity suitable for despatch to a laboratory. It also includes directions for packaging and labelling of the samples.

4.2 Freshly mixed mortars

4.2.1 General

Samples shall be obtained by taking uniformly distributed increments. The samples shall be immediately combined and mixed thoroughly to form a representative combined bulk or sub-sample. The bulk or sub-sample shall be reduced in accordance with 4.2.4.

NOTE 1. It is preferable that samples are obtained from material in motion, provided this can be carried out in safety.

NOTE 2. The number of increments and the size of the bulk or sub-sample necessary depends on the quantity of the material and its variability, and the accuracy required of the test results.

4.2.2 Apparatus

4.2.2.1 Receptacle or scoop, metal, and of not less than 1 l capacity.

4.2.2.2 Airtight containers. The containers shall be clean and dry at the commencement of the sampling operation.

4.2.3 Taking of samples

4.2.3.1 Batch mixers

Sample the mortar at the discharge point of a batch from the mixer. Take not less than 12 increments spaced evenly through the batch. Take the increments by passing the receptacle across the stream of mortar in such a manner as to collect a thoroughly representative sample of mortar.

4.2.3.2 Conveyors, pumps, etc.

Where possible, take increments at the discharge of a conveyor or pipeline. Pass the receptacle across the stream of material, if possible so as to catch the whole of the stream, until it is filled. If it is not possible to catch the whole stream at once, pass the receptacle through the stream at a uniform rate in a manner that is varied on a systematic pattern for successive increments.

Where it is not possible to sample at the discharge point of the conveyor, take increments using a scoop from the full width and thickness of the stream of material on the conveyor after stopping the conveyor. The scoop used shall sweep the surface of the conveyor.

Take not less than 12 increments at regularly spaced intervals during the passage of the whole of the quantity of the material that is being sampled. Combine the increments to form one bulk sample.

4.2.3.3 Large hoppers, bins or heaps

Sample the mortar in accordance with 4.2.3.2 only when hoppers, etc., are being filled, prepared, emptied or removed.

4.2.3.4 Small hoppers, bins or heaps

Sample the mortar by means of the scoop at regular spacings throughout the mass. Take increments from material well below the surface in at least 12 different places in the mass, distributed in a regular manner so as to ensure, when mixed, a thoroughly representative combined sub-sample.

4.2.3.5 Bulk transport vehicles

Either sample the contents of bulk transport containers during filling or emptying by the procedures given in 4.2.3.2 or, when this is not practicable, by taking sub-samples as described in 4.2.3.4.

4.2.4 Reduction of sample

Reduce the bulk sample or sub-sample(s) to a mass of not less than 15 kg by taking sufficient scoopfuls from random positions throughout the mixed material. The reduced sample shall be placed in one or more airtight containers.

NOTE. If the consistence and air content tests are to be made on a sample, arrangements should be made to carry out the tests at the point of sampling.

4.2.5 Packaging and certificate of sampling

Each sample to be despatched to a laboratory shall be placed in one or more airtight containers, and suitably labelled so that its origin can be identified at the laboratory. The sample shall be accompanied by a certificate from the person responsible for taking the sample stating that sampling was carried out in accordance with the requirements of this British Standard.

The certificate shall include as much of the following information as is appropriate:

- a) type of material;
- b) the date, time, place and method of sampling;
- c) the quantity of the batch and consignment, or the period of production represented by the sample;
- d) tests required.

4.3 Hardened mortars

4.3.1 General

Because of the variety of circumstances in which sampling of hardened mortars may be required, only general guidance is given here. Samples may be required to ascertain the following:

- a) the variability in different parts of the work.
For this purpose, sub-samples should be kept separate and the areas represented by each should be carefully recorded. Results of tests on such samples should be regarded only as representative of the mortar from the area indicated;
- b) the composition at specific points, e.g. where defects have been observed.

In this case, the location and the depths from the surface of the work represented by the sample should be recorded;

c) the average composition over a significant area of masonry, plastering, rendering, or screed.

For this purpose, it should be realized that a number of different batches may have been used, and a number of sub-samples of approximately equal mass should be taken from representative areas and combined to make a composite main sample, which is as representative of the average as is practicable. The uncertainties in doing this inevitably limit the value of such average samples and these should be recognized when interpreting results. Care should be taken to avoid taking samples for this purpose predominantly from points at which the mortar is more readily extractable, since the mortar at such points is likely to be untypical of the average composition;

d) information about adhesion.

In this case the sample should include the substrate.

4.3.2 Apparatus

4.3.2.1 Tools for cutting out the sample, such as hammer and bolster or cold chisel, or masonry or core drill.

4.3.2.2 Means of collecting representative samples, without loss of fine material.

4.3.2.3 Containers, that will prevent sample loss or contamination.

4.3.3 Size of samples

Samples for despatch to the laboratory shall each be not less than 100 g in mass. Where a number of sub-samples are combined to form one composite main sample, about the same amount of each sub-sample shall be included.

Where samples are intended to represent averages of the mortar over substantial areas of work, sub-samples shall be combined to form a composite main sample. The minimum size of sub-sample for each 10 m² and the maximum area represented by each composite main sample shall be as given in table 1.

Table 1. Representative sample sizes		
	Minimum mass of sub-sample to represent 10 m ² g	Maximum area represented by each composite main sample m ²
Brick or block work	50	50
Plastering or rendering	50	50
Floor screed	100	100

4.3.4 Taking of samples and source

4.3.4.1 Masonry mortar

Take a number of sub-samples sufficient to produce a representative quantity of mortar by removing a number of bricks or blocks from the construction. For most purposes, carefully remove the mortar from the bricks or blocks, avoiding contamination with the material of which they are composed. Take each sub-sample over the thickness of the structure of which it is intended to be representative, and record that thickness.

Where it is essential to avoid the removal of bricks or blocks, and where only chemical composition is required, take the sub-samples using a masonry drill or core drill, or by chipping away the mortar. Ensure that the sub-samples are not contaminated with the material of which the bricks or blocks are composed, and that there is no loss of the finer material.

NOTE. The use of a masonry drill for sampling brickwork mortar can lead to an unrepresentative and segregated sample. Where the mortar is pigmented and is of similar colour to the bricks, any residue from the bricks will be difficult to detect in the sample.

4.3.4.2 Plastering and rendering mortar

Take sub-samples from regularly spaced positions over the area to be examined. Where adhesion is good, cut out cores and, for most purposes, remove any adhering background material. Examine the background and record any observations.

NOTE 1. When a failure of adhesion to the background has occurred, appropriate areas may be removed with any convenient tool.

NOTE 2. When adhesion has failed between coats of mortar, separate sub-samples of each coat should be taken.

4.3.4.3 Screeding mortar

Each sub-sample shall be a coherent piece of full depth. Take the sub-samples from regularly spaced positions over the area to be examined. When a failure of adhesion to the sub-base has occurred break out appropriate sub-samples, using a hammer and cold chisel or bolster. Where adhesion is good, cut out or core the sub-samples. Examine the surface of the sub-base for the presence of grout and/or foreign matter.

4.3.5 Packaging and certificate of sampling

Each sample to be despatched to a laboratory shall be placed in one or more containers and suitably labelled so that its origin can be identified at the laboratory. The samples shall be accompanied by a certificate from the person responsible for taking the sample stating that sampling was carried out in accordance with the requirements of this British Standard.

The certificate shall include as much of the following information as appropriate:

- the date, time, place, and method of sampling;
- the location in the building of the area sampled, and the depths from the face of the structure represented by the sample;
- the state of the mortar at the time of sampling, e.g. wet or dry, soft or hard;
- such information as is available on the nature of the contiguous material, e.g. the type of brick or block, and any adhering material such as finishing plaster, paint, grout;
- reason for investigation and specified mix, if known.

5 Apparatus

5.1 General. All volumetric glassware shall have an accuracy of class B or better (where given) in the appropriate British Standard.

NOTE 1. It is assumed that a laboratory carrying out these tests will be equipped with basic apparatus such as analytical balances, beakers, volumetric flasks, pipettes, burettes, filtration apparatus, stirrers etc.

NOTE 2. Only apparatus particular to the determinations necessary to analyse hardened mortar is listed.

5.2 250 ml polypropylene beakers, conforming to BS 5404 : Part 1.

5.3 Furnace, or furnaces controlled to maintain temperatures of $(850 \pm 25)^\circ\text{C}$, $(925 \pm 25)^\circ\text{C}$ and $(1200 \pm 25)^\circ\text{C}$.

5.4 Boiling water or steam bath.

5.5 Test sieves, conforming to BS 410.

5.6 Pestle and mortar or mechanical crusher.

6 Reagents

6.1 General. Unless otherwise stated, all reagents shall be of analytical quality, and distilled or de-ionized water conforming to BS 3978 shall be used throughout the analysis. Reagents shall conform to **6.2 to 6.48**. Where reagents are prepared in the laboratory, the procedures given in clause 6 shall be followed.

NOTE 1. Proprietary reagents of analytical quality may be used.

NOTE 2. Dilutions of acids and other liquid reagents are given as $(1 + n)$, which indicates that one volume of the concentrated reagent is added to n volumes of water and mixed. Care should be taken when adding concentrated sulfuric acid to water as the solution becomes very hot and a sudden evolution of steam may cause spurting.

NOTE 3. Relative densities of liquid reagents are given using the symbol d .

Solutions of solid reagents shall be filtered if the solution is not clear.

6.2 *Acetic acid, glacial.*

6.3 *Ammonia solution, $d = 0.880$.*

6.4 *Ammonium acetate buffer.* Add, with stirring, 120 ml of acetic acid (see **6.2**) to 500 ml of water, followed by 74 ml of ammonia solution (see **6.3**). Cool, dilute to 1 l and mix.

6.5 *Ammonium acetate solution,* approximately 100 g/l. Dilute 70 ml of acetic acid (see **6.2**) to 1 l with water and add carefully 70 ml of ammonia solution (see **6.3**), mix, cool and adjust to pH 6, either with acetic acid or ammonia solution.

6.6 *Ammonium chloride solution, 1 g/l.* Dissolve 1 g of ammonium chloride in 1 l of water.

6.7 *Ammonium thiocyanate standard solution,* approximately 0.1 M. Dissolve approximately 7.6 g of ammonium thiocyanate in water and dilute to 1 l in a volumetric flask. Standardize against the silver nitrate standard solution using the iron III indicator.

6.8 *Barium chloride solution, 100 g/l.* Dissolve 100 g of barium chloride in 1 l of water.

6.9 *Bromophenol blue indicator solution.* Grind 0.1 g of bromophenol blue with about 1.5 ml of sodium hydroxide solution (approximately 0.1 M) and then dilute to 100 ml with water.

6.10 *Calcein (screened) indicator.* Mix, by grinding together, 0.2 g of calcein, 0.12 g of thymolphthalein and 20 g of potassium chloride.

6.11 *Calcium standard solution, 1.0 mg CaO/ml.* Dissolve 1.785 g of dried (150 °C) calcium carbonate in a slight excess of hydrochloric acid (1 + 4), boil to expel carbon dioxide, cool and dilute to 1 l in a volumetric flask.

6.12 *Chloroform.*

6.13 *Cupferron solution, 60 g/l.* Dissolve 3 g of cupferron in 50 ml of cold water and filter. This solution shall be freshly prepared.

6.14 *DCTA standard solution, 0.05 M.* Dissolve 18.218 g of DCTA (1,2-diaminocyclohexane tetra-acetic acid) in 500 ml of water by the progressive addition of the minimum amount of potassium hydroxide solution (250 g/l).

NOTE. Approximately 25 ml should be required.

Dilute to 1 l in a volumetric flask. Store in a polyethylene bottle. Standardize against standard zinc solution as follows. Transfer 50.0 ml of the DCTA solution to a 500 ml conical flask and add five to six drops of hydrochloric acid (see **6.18**). Add a few drops of bromophenol blue indicator solution and then add ammonium acetate buffer solution until the indicator turns blue, followed by 10 ml in excess. Add a volume of ethanol (see **6.17**) equal to the total volume of the solution, followed by 1 ml to 2 ml of dithizone indicator solution and titrate with the standard zinc solution (see **6.48**) from blue-green to the first appearance of a permanent pink colour.

6.15 *Dithizone indicator solution.* Dissolve 0.012 g of dithizone in 50 ml of ethanol (see **6.17**). This solution will keep for about 1 week.

6.16 *EDTA standard solution, 6.67 g/l.* Dissolve 6.67 g of EDTA (diaminoethanetetra-acetic acid, disodium salt) in warm water, and filter. Cool and dilute to 1 l. Store in a polyethylene bottle. Standardize against standard calcium and magnesium solution as follows:

a) Calcium.

Pipette 20.0 ml of calcium standard solution (see **6.11**) into a 500 ml conical flask. Add 10 ml of potassium hydroxide solution (see **6.38**) and dilute to about 200 ml. Add about 0.015 g of screened calcein indicator and titrate with the EDTA standard solution (see **6.16**) from fluorescent green to pink. Calculate the CaO equivalent of the EDTA standard solution in mg CaO/ml;

b) Magnesium.

Pipette 20.0 ml of magnesium standard solution (see **6.28**) into a 500 ml conical flask, add 20 drops of hydrochloric acid (see **6.18**), 20 ml of ammonia solution (see **6.3**) and dilute to about 200 ml. Add about 0.04 g of methylthymol blue complexone indicator and titrate with the EDTA standard solution (see **6.16**) from blue to colourless. Calculate the MgO equivalent of the EDTA standard solution in mg MgO/ml.

6.17 *Ethanol, 95 %.* Industrial methylated spirit.

NOTE. Ethanol may be replaced for this purpose by industrial methylated spirits, 66° O.P., conforming to BS 3591. It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1952 (SI 1952, No. 2230) [1]. It is not permissible to use duty-free ethanol, received under the provisions of the Customs and Excise Act, 1952, Section III [2], for purposes for which industrial methylated spirits is an acceptable alternative to ethanol.

6.18 *Hydrochloric acid, $d = 1.18$.*

6.19 *Dilute hydrochloric acid, (1 + 9).*

6.20 *Dilute hydrochloric acid, (1 + 49).*

6.21 *Hydrochloric acid, approximately 0.5 M.* Dilute 45 ml of hydrochloric acid (see **6.18**) to 1 l. Standardize against sodium carbonate standard solution (see **6.41**) using methyl orange as the indicator.

6.22 *Hydrofluoric acid, $d = 1.13$ or 40 % (m/m).*

6.23 *Hydrogen peroxide, 6 %.*

6.24 *Hydroxylammonium chloride solution, 100 g/l.* Dissolve 100 g of hydroxylammonium chloride in 1 l of water.

6.25 *Iron III indicator solution.* To 100 ml of cold saturated solution of ammonium ferric sulfate, add 10 ml of nitric acid (see **6.32**).

6.26 Iron standard solution, 1.0 mg Fe_2O_3 /ml. Dissolve 0.491 g of ammonium ferrous sulfate in water and add 10 ml of diluted sulfuric acid (1 + 1) and 5 ml of hydrogen peroxide (see 6.23). Boil for 15 min to decompose the excess of hydrogen peroxide. Cool and dilute to 1 l in a volumetric flask.

6.27 Iron standard solution (diluted). Dilute 50 ml of the standard iron solution to 500 ml in a volumetric flask (1 ml = 0.01 mg Fe_2O_3). Prepare this solution freshly when required.

Prepare a calibration graph of the optical density measurements of solutions of different Fe_2O_3 contents using the following procedure.

Transfer 0, 10, 20, 25, 40 and 50 ml aliquots of dilute iron standard solution to 100 ml volumetric flasks.

Add to each 2 ml of hydroxylammonium chloride solution (see 6.24), 5 ml of 1,10-phenanthroline solution (see 6.33) and 2 ml of ammonium acetate solution (see 6.5). Allow to stand for 15 min, dilute the solution in each flask to 100 ml and shake well.

Measure the optical density of the solutions against water in 10 mm cells at 510 nm or by using a colour filter in a suitable instrument.

NOTE. An Ilford 603 filter has been found to be suitable.

6.28 Magnesium standard solution, 0.1 mg MgO /ml. Wash the metal in hydrochloric acid (see 6.19) to remove any oxide film, then with water and finally with alcohol followed by ether. Dissolve 0.603 g of the oxide-free metal in a slight excess of hydrochloric acid (see 6.19), cool and dilute to 1 l in a volumetric flask.

6.29 Methyl orange indicator solution. Dissolve 0.05 g of methyl orange in 100 ml of hot water, cool and filter.

6.30 Methylthymol blue complexone indicator. Mix, by grinding together, 0.2 g of methylthymol blue complexone and 20 g of potassium nitrate.

6.31 Naphthol green B solution, 1 g/l. Dissolve 0.1 g of naphthol green B in 100 ml of water.

6.32 Nitric acid, $d = 1.42$.

6.33 1,10-phenanthroline solution, 10 g/l. Prepare enough solution for immediate use at a concentration of 0.1 g 1,10-phenanthroline hydrate in 10 ml of acetic acid (1 + 1).

6.34 Phenolphthalein indicator solution. Dissolve 1 g of phenolphthalein in 100 ml of ethanol (see 6.17).

6.35 Polyacrylamide solution, 1 g/l. Add 0.1 g of polyacrylamide, relative molecular mass about 5×10^6 , to 100 ml of water. Stir using a mechanical stirrer until dissolved.

6.36 Polyethylene oxide solution, 2.5 g/l. Add 0.5 g of polyethylene oxide, relative molecular mass less than 1×10^6 , to 200 ml of water slowly, stirring, preferably using a mechanical stirrer, until dissolved. Discard after two weeks.

6.37 Potassium hydrogen sulfate, solid.

6.38 Potassium hydroxide solution, 250 g/l. Dissolve 250 g of potassium hydroxide in 1 l of water.

6.39 Silver nitrate standard solution, 0.1 M. Either dry powdered silver nitrate at 150°C for 2 h, cool in a desiccator, dissolve 16.989 g in water and dilute to 1 l.

Alternatively, dissolve 10.788 g of pure silver metal in the minimum amount of nitric acid (see 6.32) and dilute to 1 l.

6.40 Sodium carbonate solution, 100 g/l. Dissolve 100 g of anhydrous sodium carbonate in 1 l of water.

6.41 Sodium carbonate standard solution, 0.25 M. Dissolve 6.625 g of anhydrous sodium carbonate, dried at 150°C , in water and dilute to 250 ml in a volumetric flask.

6.42 Sodium hydroxide standard solution, approximately 0.5 M. Dissolve 20 g of sodium hydroxide pellets in 250 ml of freshly boiled, cooled, distilled water, and cool and dilute to 1 l. Standardize against the hydrochloric acid solution (see 6.21) using phenolphthalein as indicator.

6.43 Sugar, granulated.

6.44 Sulfuric acid, $d = 1.84$.

6.45 Triethanolamine, (1 + 1).

6.46 Triethanolamine, (1 + 4).

6.47 Trimethyl hexanol (nonyl alcohol).

6.48 Zinc standard solution, 0.05 M. Wash the metal in hydrochloric acid (see 6.19) to remove any oxide film, then with water and finally with alcohol followed by ether. Dissolve 3.269 g of the oxide free metal in 10 ml of hydrochloric acid (see 6.18) and about 50 ml of water. Cool and dilute to 1 l in a volumetric flask. A 1 ml volume of this solution is equivalent to 2.55 mg Al_2O_3 .

NOTE. Zinc pellets are a convenient form of the metal.