

Methods of testing

Mortars, screeds and plasters

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The organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

Brick Development Association	Concrete Block Association
British Ceramic Research Association	Mortar Producers' Association Ltd
British Civil Engineering Test Equipment Manufacturers' Association	National Federation of Plastering Contractors
Calcium Silicate Brick Association Limited	Plasterers' Craft Guild

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Foreword

The publication in 1970, as BS 4551, of the first British Standard methods of testing mortars enabled the properties of mortars to be studied systematically and the results obtained from various sources to be compared. After ten years use, some modifications and extensions to methods and interpretation of results are necessary. The standard has been extended to include mortars made with masonry cement, gypsum-based plasters and cement : sand screeding mortars and the title amended accordingly. The specification for mortar-testing sand has been omitted following the publication of BS 4550-5 of which is referred to, and new analytical methods have been included.

The original standard contained sufficient instructions for sampling freshly mixed mortars and these have been included in the present edition. Although recommendations for sampling hardened mortars, etc., were given in an appendix to the standard these were considered to be insufficient and more details are now included in this revision. The need for completing and attaching a certificate of sampling is stressed to all who are concerned with the testing of building mortars and plasters.

The standard is now divided into three sections: General; Methods of chemical analysis of mortars and the interpretation of the results; and Physical tests such as consistency, water retention, flow etc. Clause 7, which describes the procedure for interpreting the results, if used universally, will ensure more consistent interpretations. However, it is impossible to relate the analytical results to volume proportions of a mortar mix with any great accuracy and therefore use of the dry mass basis has been introduced, which it is recommended should eventually replace the use of volume proportions.

Although it is desirable that tests suitable for site use should be included in this standard, insufficient evidence is available to date for these to be included in the present revision. The testing of bricks/blocks/stone and mortars and plasters together is considered to be the most satisfactory approach for determining the quality of masonry and in BS 5628-1 this principle is adopted. Consideration is also being given to the methods in the Australian Standard AS 1640¹⁾ and to the work of Ryder²⁾. Recently Pye and Warlow³⁾ have developed an apparatus for assessing the soundness of some dense floor screeds and if this method proves to be satisfactory, it will be considered for inclusion in future standards.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii pages 1 to 40, 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⁾ AS 1640:1974 "Brickwork in buildings, known as the SAA brickwork code". Standards Association of Australia.

²⁾ Ryder, J F. "The use of small brickwork panels for testing mortars". *Transactions of the British Ceramic Society*, Vol. 62, No. 8, August 1963, pp. 615-622.

³⁾ Pye P W and Warlow W J. "A method of assessing the soundness of some dense floor screeds". *Document PD 70/78 Building Research Establishment*, 1978.

Section 1. General

0 Introduction

Technical changes have been made in this revision, affecting, in the main, the chemical analysis and the physical testing of mortars, screeds and plasters.

Normally chemical analysis is required because a failure has occurred and the analysis will generally indicate the cause of the failure. Soon after the publication of the original standard it was found that the chemical determination of soluble silica gave consistently low results for mortars with high cement contents. An alternative method was proposed, the accuracy of which was confirmed by co-operative testing between five laboratories, and this method has been included.

It has been shown⁴⁾ that the properties of lime-based mortars are related to the actual quantity of calcium hydroxide present. A greater mass of a semi-hydraulic lime is required to produce the equivalent working properties of a pure high calcium lime. The soluble silica content of hydraulic or semi-hydraulic limes increases the strength of mortars and therefore the calculated Portland cement content of mortars containing such limes will be slightly increased. Thus throughout the interpretation of results, the actual pure calcium hydroxide content is calculated. Similarly the properties of gypsum-based plasters are proportional to the amount of pure calcium sulphate present, and hence the composition is calculated as the pure calcium sulphate.

All analysed samples will have varying degrees of hydration, carbonation, etc., and, to eliminate the variation of the calculated compositions of mortars, screeds and plasters, the results are corrected to the dry mass of the original ingredients.

Four general changes have been made in the physical testing section. The first is standardizing the proportions of each type of mix. It is known that well graded aggregates have a higher bulk density than poorly graded aggregates and therefore the mass of binders is less for the well graded than the poorly graded. To remove all controversy regarding determined bulk densities of raw materials fixed masses are given for all types of mix. The second change from the original standard is that site or plant mixed materials are tested as received without correcting the consistency as determined by the dropping ball test to a standard 10 mm. The dropping ball test is made and the results reported on the consistence as received. Thirdly, it has been found that the grading recommended in the original standard for the standard sand mix was too coarse and produced an unworkable mix which shed water when used as the control for the stiffening rate test. The grading now recommended helps to eliminate these problems. The fourth change is that the use of suction in the moulding of test specimens for strength testing has been deleted. It was found that the results obtained by this procedure were of limited application because the suction used was not typical of many bricks, blocks or masonry.

The bulk density of the hardened sample of mortar should be determined wherever possible. A low bulk density could indicate either excessive air content or the presence of a poorly graded sand.

1 Scope

This British Standard specifies the methods of sampling, analysis, preparation and testing of mortars for bricklaying, screeding, plastering and rendering.

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

2 References

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

3 Definitions

For the purposes of this British Standard the definitions given in BS 2787 and BS 4049 apply.

⁴⁾ Gillard R and Lee H N. "Testing of building mortars using the new British Standard methods". *ASTM Special Technical Publication STP 472*, 1969, pp 82-113.

4 Sampling

4.1 Introduction. 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 (preferably from material in motion, provided this can be carried out in safety), and mixed thoroughly to form a combined bulk sample. The number of increments and size of bulk sample necessary depends on the quality of the material and its variability and the accuracy required of the test results. The bulk sample shall be reduced in accordance with 4.2.4.

4.2.2 Apparatus. According to the method being used, the apparatus required is either a metal receptacle or a scoop of not less than 1 litre capacity, and airtight containers which shall be clean and dry at the commencement of the sampling operation.

4.2.3 Taking of samples

4.2.3.1 Batch mixers. The mortar shall be sampled at the discharge point of a batch from the mixer. Not less than twelve increments spaced evenly through the batch shall be taken at the discharge point of the mixer. The increments shall be taken 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. It is preferable to take increments at the discharge of a conveyor or pipeline. This shall be done by passing 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, the receptacle shall be passed 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 conveyor, increments shall be taken 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.

Not less than twelve increments shall be taken at regularly spaced intervals during the passage of the whole of the quantity of the material that is being sampled. The increments shall be combined to form one bulk sample and treated as in 4.2.4.

4.2.3.3 Large hoppers, bins or heaps. It is essential that material be sampled when hoppers, etc., are being filled, prepared, emptied or removed, when the directions given in 4.2.3.2 shall be followed.

4.2.3.4 Small hoppers, bins or heaps. The material shall be sampled by means of the scoop at regular spacings throughout the mass. Increments shall be taken from material well below the surface in at least twelve different places in the mass, distributed in a regular manner, so as to ensure, when mixed, a thoroughly representative combined sample.

4.2.3.5 Bulk transport vehicles. The contents of bulk transport containers shall either be sampled 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 bulk sample. The increments or sub-samples taken in accordance with any of the methods described shall immediately be combined and thoroughly mixed and reduced to a sample of not less than 15 kg⁵⁾ 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 in this clause. Samples may be required to ascertain the following.

⁵⁾ Where the sample is required solely for chemical analysis, only 1 kg is required.

4.3.1.1 *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.

4.3.1.2 *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.

4.3.1.3 *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 shall 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.

4.3.1.4 *Information about adhesion.* In this case the sample shall include the substrate.

4.3.2 *Apparatus.* The following apparatus is required.

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 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, the minimum size of sub-sample for each 10 m² and the maximum area represented by each main sample shall be as follows.

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

4.3.4 *Taking of samples and source*

4.3.4.1 *Masonry mortar.* The sub-samples shall each be obtained by removing a number of bricks or blocks from the construction, sufficient to produce a representative quantity of mortar. For most purposes the mortar shall be carefully removed from the bricks or blocks, avoiding contamination with the material of which they are composed. The sample shall be taken over the thickness of the structure of which it is intended to be representative, and that thickness shall be recorded.

Where it is essential to avoid the removal of bricks or blocks, and where only chemical composition is required, the sample may be obtained by use of a masonry drill or core drill or by chipping away the mortar, care being taken to ensure that the 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.

4.3.4.2 *Plastering and rendering mortar.* Samples shall be taken from regularly spaced positions over the area to be examined. When a failure of adhesion to the background has occurred, appropriate areas may be removed with any convenient tool. Where adhesion is good, cores shall be cut out and, for most purposes, any adhering background material removed. When adhesion has failed between coats, separate samples of each coat should be taken. The background shall be examined and any observations recorded.

4.3.4.3 *Screeding mortar.* Each sub-sample shall be a coherent piece of full depth and shall be taken from regularly spaced positions over the area to be examined. When a failure of adhesion to the sub-base has occurred appropriate samples may be broken out, using a hammer and cold chisel or bolster. Where adhesion is good samples may be cut out or cored. The surface of the sub-base shall be examined 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:

- a) the date, time, place, and method of sampling;

- b) the location in the building of the area sampled, and the depths from the face of the structure represented by the sample;
- c) the state of the mortar at the time of sampling, e.g. wet or dry, soft or hard;
- d) 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;
- e) reason for investigation and specified mix, if known.

Section 2. Chemical testing

5 Laboratory preparation of samples for chemical analysis

5.1 Introduction. This clause describes procedures for laboratory preparation of samples for the analysis of fresh and hardened mortars, screeds and plasters.

Whenever it is necessary to prepare a test sample from a larger quantity received at the laboratory, subdivide the sample by coning and quartering or by means of a sample divider such as a riffle box. All test sieves used shall comply with the requirements of BS 410.

5.2 Freshly mixed lime: sand for mortars.

Thoroughly mix each sample and reduce to about 250 g, then place in a wide-mouthed jar and enclose with an airtight cap. Immediately before starting each of the tests in 6.3.1, remix the reduced sample with a spatula.

5.3 Freshly mixed cement-based mortars. By the time the sample has arrived in the laboratory hardening has normally occurred. The sample shall be dried immediately on receipt at 105 ± 5 °C to constant mass, thus preventing the complete setting of the mortar and thereby facilitating subsequent reduction of the sample.

Crush the dried sample to pass a 10 mm test sieve, avoiding, as far as possible, fracture of aggregate particles and reduce to 250 g. The sample shall then be treated as in 5.5.

5.4 Freshly mixed gypsum-based plasters. As gypsum-based backing plasters set reasonably quickly the sampling shall follow the method described in 5.5.

5.5 Hardened mortars, screeds and plasters.

The preparation often involves the separation by suitable means of various layers of a multi-layer system and freeing them from backing materials, decorative finishes and contaminants. Difficulty sometimes arises in finding the interface between what are reputedly two layers of mortar or plaster. In such cases, take samples as near to the outside faces of the composite layer as possible or, if the composite layer is too thin to allow this analyse it as one layer, making an appropriate note to the effect on the report. If, because of the separation of layers, any such sub-sample is less than 100 g only a chemical analysis shall be undertaken.

Measure the mortar, screed or plaster sample to determine the average thickness. In the case of screeds, examine the bottom surface for foreign matter and grout. Examine the uniformity of piece samples to establish efficiency of mixing, and note the presence of nodules of sand, etc. Report all this information with the other tests on the sample.

Crush the sample to pass a 10 mm test sieve, avoiding as far as possible fracture of aggregate particles, and reduce to 250 g. Dry this sample at 105 ± 5 °C in the case of cement-based materials, or at 40 ± 2 °C for gypsum-based materials, to constant mass and place in a jar and enclose with an airtight cap.

If aggregate grading is required, sufficient test samples, each of about 50 g mass, shall be taken by an efficient sampling method.

If chemical analysis is required, crush the remainder of the sample to pass a 150 µm test sieve avoiding as far as possible excessive particle size reduction. This shall be the analytical sample.

If suitable mechanical grinding equipment is not available, use the following procedure to obtain the analytical sample. Crush the remainder of the sample to pass a 2.4 mm test sieve. Grind a representative sub-sample of mass not less than 30 g to pass a 600 µm test sieve. Mix the ground sub-sample well and from this take a further sub-sample of mass at least 12 g and grind to pass a 150 µm test sieve. This shall be the analytical sample. Reserve the remainder of the 600 µm sample for chloride determination if required.

NOTE Metallic iron introduced during the grinding procedure should be removed with a magnet before any chemical analysis is commenced.

6 Chemical analysis and aggregate grading

6.1 Introduction. This clause describes a procedure for determining the grading of the aggregate used in the mix, but it should be stressed that, where aggregates contain acid-soluble material, the result will be inaccurate. It is also possible that as test methods differ from those given in BS 812, slightly different results may be obtained.

This clause specifies a technique for the determination of soluble silica and it is essential that this method be followed exactly. Recommended methods are given for the determination of other radicals but other methods may be used provided they are of equivalent accuracy. Duplicate analyses should normally be made, but single analyses may suffice when a series of samples from a single source is being analysed.

Where mix proportions only are required, the analysis need not be continued beyond the stage described in 6.3.2.4 or, in the case of gypsum-based materials, 6.3.2.5.

6.2 Reagents. Unless otherwise stated, all reagents shall be of analytical reagent quality, and distilled or de-ionized water in accordance with BS 3978 shall be used throughout the analysis.

NOTE 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 sulphuric acid to water as the solution becomes very hot and a sudden evolution of steam may cause spurting.

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

Prepare the reagents by the following methods, although proprietary reagents of proven quality may be used.

Acetic acid. Glacial.

Ammonia solution. *d* = 0.880.

Ammonium acetate buffer. Add, with stirring, 120 ml of acetic acid (glacial) to 500 ml of water, followed by 74 ml of ammonia solution (*d* = 0.880). Cool, dilute to 1 litre and mix.

Ammonium acetate solution (approximately 100 g/litre). Dilute 70 ml of acetic acid (glacial) to 1 litre with water and add carefully 70 ml of ammonia solution (*d* = 0.880), mix, cool and adjust to pH6, either with acetic acid or ammonia solution.

Ammonium chloride solution (1 g/litre). Dissolve 1 g of ammonium chloride in 1 litre of water.

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

Barium chloride solution (100 g/litre). Dissolve 100 g of barium chloride in 1 litre of water.

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.

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

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 litre in a volumetric flask.

Chloroform.

Cupferron solution (60 g/litre). Dissolve 3 g of cupferron in 50 ml of cold water and filter. This solution must be freshly prepared.

DCTA standard solution (0.05 M). Dissolve 18.2175 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/litre) (approximately 25 ml should be required). Dilute to 1 litre 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 5 to 6 drops of hydrochloric acid (*d* = 1.18). Add a few drops of bromophenol blue indicator solution and then add ammonium acetate buffer solution until the indicator turns blue, follow by 10 ml in excess.

Add a volume of ethanol (95 %) equal to the total volume of the solution, follow by 1 ml to 2 ml of dithizone indicator solution and titrate with the standard zinc solution (0.05 M) from blue-green to the first appearance of a permanent pink colour.

Dithizone indicator solution. Dissolve 0.0125 g of dithizone in 50 ml of ethanol (95 %). This solution will keep for about 1 week.

EDTA solution (5 g/litre). Dissolve 5 g of EDTA (diaminoethane tetra-acetic acid, disodium salt) in warm water, and filter, cool and dilute to 1 litre. Store in a polyethylene bottle. Standardize against standard calcium and magnesium solutions as follows:

Calcium. Pipette 25.0 ml of standard calcium solution (1.0 mg (CaO)/ml) into a 500 ml conical flask, add 10 ml of potassium hydroxide solution (250 g/litre) and dilute to about 200 ml. Add about 0.015 g of screened calcein indicator and titrate with the EDTA solution (5 g/litre) from fluorescent green to pink. Calculate the CaO equivalent of the EDTA solution in mg (CaO)/ml.

Magnesium. Pipette 25.0 ml of standard magnesium solution (1.0 mg (MgO)/ml) into a 500 ml conical flask, add 20 drops of hydrochloric acid ($d = 1.18$), 20 ml of ammonia solution ($d = 0.880$) and dilute to about 200 ml. Add about 0.04 g of methylthymol blue complexone indicator and titrate with the EDTA solution (5 g/litre) from blue to colourless. Calculate the MgO equivalent of the EDTA solution in mg (MgO)/ml.

Ethanol (95 %). Industrial methylated spirit.⁶⁾

Hydrochloric acid. $d = 1.18$.

Hydrochloric acid (approximately 0.5 M).

Dilute 45 ml of hydrochloric acid ($d = 1.18$) to 1 litre. Standardize against sodium carbonate standard solution (0.25 M) using methyl orange as indicator.

Hydrofluoric acid. $d = 1.13$ or 40 % m/m .

Hydrogen peroxide (6 %).

Hydroxylammonium chloride solution (100 g/litre). Dissolve 100 g of hydroxylammonium chloride in 1 litre of water.

Iron III indicator solution. To 100 ml of cold saturated solution of ammonium ferric sulphate add 10 ml of nitric acid ($d = 1.42$).

Iron standard solution (0.1 mg (Fe₂O₃)/ml).

Dissolve 0.4911 g of ammonium ferrous sulphate in water and add 10 ml of diluted sulphuric acid (1 + 1) and 5 ml of hydrogen peroxide (6 %). Boil for 15 min to decompose the excess of hydrogen peroxide. Cool and dilute to 1 litre in a volumetric flask.

Iron standard solution (diluted). Dilute 50 ml of the standard iron solution to 500 ml in a volumetric flask (1 ml = 0.01 mg Fe₂O₃). Prepare this solution freshly when required.

Prepare a calibration graph of the optical density measurements of solutions of different Fe₂O₃ contents using the following procedure.

Transfer 0, 10, 20, 25, 40 and 50 ml aliquots of the dilute iron standard solution to 100 ml volumetric flasks. Add to each 2 ml of hydroxylammonium chloride solution (100 g/litre), 5 ml of 1,10-phenanthroline solution (10 g/litre) and 2 ml of ammonium acetate solution (approx. 100 g/litre). 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 filter⁷⁾ in a suitable instrument.

The colour is stable between 15 min and 75 min after the addition of the ammonium acetate solution.

Magnesium standard solution (1.0 mg (MgO)/ml). Wash the metal in hydrochloric acid (1 + 9) to remove any oxide film, then with water and finally with alcohol followed by ether. Dissolve 0.6032 g of the oxide-free metal in a slight excess of hydrochloric acid (1 + 9), cool and dilute to 1 litre in a volumetric flask.

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

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

Naphthol green B solution (1 g/litre). Dissolve 0.1 g of naphthol green B in 100 ml of water.

Nitric acid. $d = 1.42$.

1,10-phenanthroline solution (10 g/litre). 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).

Phenolphthalein indicator solution. Dissolve 1 g of phenolphthalein in 100 ml of ethanol (95 %).

Polyacrylamide solution (1 g/litre). Add 0.1 g of polyacrylamide, molecular weight about 5×10^6 , to 100 ml of water. Stir using a mechanical stirrer until dissolved.

⁶⁾ Ethanol may be replaced for this purpose by industrial methylated spirits, 66 degrees O.P., complying with the requirements of BS 3591. It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1952 (S.I. 1952, No. 2230). It is not permissible to use duty-free ethanol, received under the provisions of the Customs and Excise Act, 1952, Section III, for purposes for which industrial methylated spirits is an acceptable alternative to ethanol.

⁷⁾ An Ilford 603 filter has been found to be suitable.

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

Potassium hydrogen sulphate. Solid.

Potassium hydroxide solution (250 g/litre).

Dissolve 250 g of potassium hydroxide in 1 litre of water.

Silver nitrate standard solution (0.1 M). Dry powdered silver nitrate at 150 °C for 2 h. Cool in a desiccator and dissolve 16.989 g in water and dilute to 1 litre.

Alternatively, dissolve 10.788 g of pure silver metal in the minimum amount of nitric acid ($d = 1.42$) and dilute to 1 litre.

Sodium carbonate solution (100 g/litre).

Dissolve 100 g of anhydrous sodium carbonate in 1 litre of water.

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.

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 litre. Standardize against the hydrochloric acid solution (approximately 0.5 M) using phenolphthalein as indicator.

Sugar. Granulated.

Sulphuric acid. $d = 1.84$.

Triethanolamine.

Trimethyl hexanol (nonyl alcohol).

Zinc standard solution (0.05 M). Wash the metal in hydrochloric acid (1 + 9) to remove any oxide film, then with water and finally with alcohol followed by, ether. Dissolve 3.2685 g of the oxide free metal in 10 ml of hydrochloric acid ($d = 1.18$) and about 50 ml of water. Cool and dilute to 1 litre in a volumetric flask. 1 ml of this solution is equivalent to 2.55 mg Al_2O_3 .

NOTE Zinc pellets are a convenient form of the metal.

6.3 Type of sample

6.3.1 Ready-mixed lime: sand for mortars

6.3.1.1 General. In this section the methods apply only to mortars consisting of lime and fine aggregate. Attention is drawn to the practical limitation of the method to such mortars that have not suffered atmospheric carbonation. Such carbonation can occur either to the mortar on a building site or to the sample if it is not kept in a completely airtight container. If carbonation has occurred then 6.3.1.3 gives only a *minimum* value for the available lime content and this shall be supplemented by a determination of the total lime content (see 6.3.1.4) to the *maximum* amount of lime that *could* have been present. It should be recognized that this maximum value will include any calcium compounds that may have been present in the sand used.

6.3.1.2 Free water content. Weigh 10 ± 0.1 g of the sample (see 5.2) into a dry preweighed dish and dry at 105 ± 5 °C to constant mass.

Calculate the content of free water to the nearest 0.5 % from the following formula:

$$\% \text{ water (wet mass basis)} = \frac{\text{Loss in mass (g)} \times 100}{\text{Mass of test sample (g)}}$$

6.3.1.3 Available lime content. Weigh 5 ± 0.05 g of the sample (see 5.2) into a 250 ml flask containing 20 ml to 30 ml of water and immediately stopper the flask loosely with a rubber stopper. Disperse the sample thoroughly in the water by a swirling motion, loosen the stopper, heat to boiling and simmer for 2 min. Remove the stopper completely, add about 150 ml of water at room temperature and then 15 g of granulated sugar. Stopper the flask, shake vigorously at intervals for 5 min and allow to stand for at least 30 min but not longer than 1 h.

NOTE Samples that are known to have had, or appear to have had, pigments incorporated should be filtered rapidly with the aid of suction through a Buchner funnel fitted with a medium filter paper⁸⁾ and the filtrate collected in a Buchner flask and titrated immediately to avoid carbonation.

Add five drops of phenolphthalein indicator solution, wash down the stopper and sides of the flask with water and, while shaking or stirring continuously, titrate as rapidly as possible with 0.5 M standard hydrochloric acid solution until the first complete disappearance of the pink colour. Record the volume of acid used and ignore any return of the pink colour. Calculate the content of available lime to the nearest 0.1 % from the following formulae:

$$\% \text{ available lime (as \% Ca(OH)}_2 \text{ by wet mass)}$$

⁸⁾ No.40 Whatman or similar filter paper has been found to be suitable.

$$= \frac{3.7 VX}{M}$$

where

V is the volume of HCl solution required (in ml)

X is the molarity of HCl solution

M is the mass of test sample (in g)

% available lime (as % Ca(OH)_2 by dry mass)

$$= \frac{\% \text{ available lime by wet mass} \times 100}{100 - \% \text{ water by wet mass}}$$

6.3.1.4 Total lime content. Weigh 5 ± 0.05 g of the test sample into a 400 ml beaker and disperse with about 50 ml water. Add 10 ml hydrochloric acid ($d = 1.18$) and mix thoroughly. Add about 100 ml water, heat to boiling and simmer for 5 min. Filter through a coarse hardened filter paper⁹⁾ and wash the residue with hot water. Adjust the volume of the filtrate and washings, cooled to room temperature, to 500 ml in a volumetric flask. Mix thoroughly and transfer by pipette 10 ml to a 500 ml conical flask. Add 5 ml triethanolamine solution (1 + 1) and 10 ml potassium hydroxide solution (250 g/litre) and dilute to approximately 200 ml with water. Add about 0.15 g screened calcein indicator (fluorescein complexone) and, while stirring, titrate against a black background using standard EDTA (5 g/litre). The colour change is from fluorescent green to pink-purple at the end point.

Calculate the total lime content to the nearest 0.1 % from the following formula.

% total lime (as Ca(OH)_2 by wet mass)

$$= \text{titre of EDTA} \times \frac{74.1}{56.08} \times \frac{5 \times m}{\text{sample mass (g)}}$$

where

m is the CaO equivalent of EDTA (in mg).

NOTE This value represents the maximum amount of lime present and can be corrected for calcium compounds present in the sand used if they are reliably known.

6.3.1.5 Sand, silt and clay content and sand grading. Weigh a quantity, M , of the wet sample (see 5.2) equivalent to 100 g dried mortar and calculated to the nearest 0.1 g from the formula

$$M = \frac{100 \times 100}{100 - \% \text{ free water by wet mass}}$$

Place this sample in a container of sufficient size so that it can be covered with water and agitated without loss of any part of the sample or water. Add enough water to cover the sample. Agitate the contents of the container vigorously and immediately pour the wash water over the top sieve of a nest of test sieves with the following apertures 5.0 mm, 2.36 mm, 1.18 mm, 600 μm , 300 μm , 150 μm and 75 μm . The sieves shall be previously wetted on both sides and shall be arranged with the coarsest sieve on top and the finest sieve at the bottom.

The agitation shall be sufficiently vigorous to result in the complete separation from the coarse particles of all particles finer than the 75 μm test sieve and to bring the fine material into suspension in order that it will be removed by decantation of the wash water. Take care to avoid, as far as possible, decantation of the coarser particles of the sample. Repeat until the wash water is clear.

Transfer all the material in the container on to the top (coarse) sieve and wash thoroughly with a jet of water. Similarly wash the material through the subsequent sieves. Dry the sieves and residues in an oven at 105 ± 5 °C. After cooling to room temperature, brush the residue from each sieve and weigh each residue separately to obtain the sand grading. Calculate the sieve analysis of the sand as described in BS 812.

Calculate the content of (lime+silt+clay) to the nearest 0.1 % from the following formula.

% (lime + silt + clay) by dry mass = $100 - R$

where

R is the total mass (in g) of dried residues on the test sieves.

Using the available lime content of the mortar (as determined in 6.3.3), calculate the respective contents of lime, (silt + clay), and sand to the nearest 0.1 % from the following formulae.

% lime by dry mass = % available lime
(as % Ca(OH)_2 by dry mass)

% (silt + clay) by dry mass =
% (lime + silt + clay) – % lime

% sand by dry mass = $100 - \% \text{ lime}$.

NOTE It is assumed that the lime used in the mortar consists of pure Ca(OH)_2 in the dry state and that it has all passed the 75 μm test sieve. These assumptions may not be true for all limes and appropriate corrections should be made where the relevant data are known.

6.3.2 Freshly mixed and hardened cement-based mortars, screeds and plasters

⁹⁾ Whatman 541 has been found to be suitable.

6.3.2.1 General. This subclause specifies a method for the extraction and determination of soluble silica and recommends methods for other oxides. When samples of the mortar constituents are available the sand shall be analysed by the same method as the mortar. Particular care shall be taken to ensure that the extraction and measurement of soluble silica are identical for both mortar and sand. Cements and limes shall be analysed by the methods described in the appropriate British Standard.

When samples of the constituents are not available, the analysis of the mortar may lead to inaccurate assessments of mortar mix proportions and other properties. The formulae given for the calculations of oxide contents are based on the stock test solution (see 6.3.2.4) having a volume of 500 ml and on the use of the recommended aliquots. If other volumes or aliquots are used the formulae will need modification.

6.3.2.2 Sand, silt and clay content, sand grading. Transfer the 50 g sample (see 5.5), accurately weighed, to a 1 litre beaker, add 500 ml hydrochloric acid (1 + 9) and heat to about 50 °C. Maintain at this temperature, with occasional stirring, until disintegration appears to be complete. Remove from the source of heat and add, whilst stirring, 5 ml polyethylene oxide solution (2.5 g/litre), allow to settle and decant the liquid through a preweighed hardened ashless filter paper.¹⁰⁾ Wash the sand remaining in the beaker by decantation three times with about 200 ml water pouring the washings through the same paper. Wash any residue on the paper back into the original beaker, dilute to 100 ml with water and add 100 ml sodium carbonate solution (100 g/litre). Simmer gently for 15 min taking care to avoid loss by frothing. Transfer the contents through a small 75 µm test sieve in accordance with BS 410 into the original paper. Wash six times with ammonium chloride solution (1 g/litre), twice with hydrochloric acid (1 + 9) and twice with hot water. Dry the sieve residue at 105 ± 5 °C, weigh and report as sand. A sieve analysis using the sieves specified in BS 1198, BS 1199 and BS 1200 may be made on this sand according to the method described in BS 812. Dry at 105 ± 5 °C the material remaining on the filter paper, weigh and report this as silt and clay.

NOTE Where the aggregates contain acid-soluble material, the sand grading test is not accurate.

6.3.2.3 Insoluble residue and soluble silica.

Weigh $5 \pm 0.005 \text{ g}^{11)}$ of the analytical sample as prepared in 5.5 into a 250 ml polypropylene beaker and carefully add 100 ml of hydrochloric acid (1 + 9). Using a magnetic stirrer with plastic covered magnetic follower, or other suitable mechanical stirrer, stir the suspension at room temperature for 20 min. Allow to settle and decant the liquid through a preweighed hardened ashless paper.¹⁰⁾

NOTE The rate of filtration is improved by adding 2 ml of polyacrylamide solution (1 g/litre) to the acid extraction just before the completion of the 20 min stirring. The subsequent determinations based on the filtered extract are not affected.

Wash the residue in the beaker with three 25 ml portions of hydrochloric acid (1 + 49), pouring the washings through the same filter paper. Retain the filtrate.

Wash any residue on the filter paper back into the original beaker, dilute to 50 ml with water and add 50 ml sodium carbonate solution (100 g/litre). Place the beaker on a boiling water or steam bath and maintain the contents of the beaker at a temperature just below boiling for 15 min, stirring occasionally with a plastic covered rod.

Transfer the contents of the beaker into the same filter paper. Wash six times with ammonium chloride solution (1 g/litre), twice with hydrochloric acid (1 + 49) and twice with hot water. The second filtrate may be collected in a separate beaker and then carefully combined with the acid filtrate. If the second filtrate is collected in the beaker already containing the acid filtrate, stir frequently to avoid the formation of a precipitate that is difficult to dissolve again. Add 10 ml hydrochloric acid ($d = 1.18$) to the combined filtrates.

Dry the filter paper and residue to constant mass at 105 ± 5 °C and report as insoluble residue.

$$\% \text{ insoluble residue} = \frac{\text{mass of dried residue} \times 100}{\text{sample mass}}$$

NOTE 1 If the sample contains insoluble combustible material (e.g. carbon black), ignite the dried paper at 950 ± 5 °C in a preweighed crucible and reweigh. Report the loss in mass as combustible material.

¹⁰⁾ No. 540 Whatman or similar filter paper has been found to be suitable.

¹¹⁾ When low cement contents are expected, a sample mass of $10 \pm 0.005 \text{ g}$ can be taken at the analyst's discretion.

Evaporate the combined filtrates and washings to a volume of about 300 ml by boiling and then transfer the contents of the beaker to an evaporating basin. Ensure complete transfer by washing the beaker with water and adding the washings to the basin. Evaporate the contents of the evaporating basin on a boiling water or steam bath until not more than approximately 5 ml remain. Add 10 ml hydrochloric acid (1 + 1), half of an ashless filter tablet and 5 ml polyethylene oxide solution (2.5 g/litre) stirring thoroughly during and after each addition. Stand for 5 min and filter through a medium ashless filter paper¹²⁾. Quantitatively transfer all the solids to the filter and wash thoroughly with hot water.

Place the filter paper containing the precipitate in a pre-weighed platinum crucible and ignite, slowly at first until all the carbon is burnt off, and then at $1\,200 \pm 25\text{ }^{\circ}\text{C}$ to constant mass. Weigh as soluble silica.

$$\% \text{ soluble silica} = \frac{\text{mass of ignited residue} \times 100}{\text{sample mass}}$$

Adjust the volume of the filtrate and washings to 500 ml (but see note 2) in a graduated flask and use this prepared solution for other determinations as appropriate. This solution is referred to below as the stock test solution.

NOTE 2 The purity of the ignited silica precipitate can be checked by treatment with hydrofluoric acid in a fume cupboard. If iron and aluminium are to be subsequently determined in the filtrate, the ignited silica precipitate is evaporated, in the crucible, with 10 ml hydrofluoric acid containing five drops of sulphuric acid (1 + 1). Ignite the residue and weigh as before. Fuse the residue in the crucible with 0.5 g to 2 g potassium hydrogen sulphate and dissolve the cooled melt in hot water containing a few drops HCl and add to the main filtrate before diluting to 500 ml. In this case the stock test solution cannot be used for the determination of sulphur trioxide.

NOTE 3 In the procedures for CaO, SO₃, Fe₂O₃, Al₂O₃ and MgO, the aliquots proposed are for a 5 g sample. If a 10 g sample is used, the necessary adjustment will need to be made to provide the equivalent aliquots.

6.3.2.4 Calcium oxide (CaO). Pipette 5 ml of the "stock test solution" into a 500 ml conical flask and add 5 ml triethanolamine solution (1 + 1) and 10 ml of potassium hydroxide solution (250 g/litre). Dilute to approximately 200 ml with water and add about 0.15 g screened calcein indicator. Swirl to ensure complete homogeneity and titrate against a black background using EDTA (5 g/litre) from a semi-micro burette. The colour change is from fluorescent green to pink-purple at the end point.

$$\% \text{ CaO} = \frac{\text{Titre EDTA used} \times \text{CaO equivalent in mg} \times 10}{\text{sample mass}}$$

6.3.2.5 Sulphur trioxide (SO₃). Pipette 100 ml of the "stock test solution" (but see note 2 in 6.3.2.3) into a 400 ml beaker and dilute with water to approximately 250 ml. Bring to the boil and add 10 ml of hot barium chloride (100 g/litre) dropwise from a pipette whilst stirring continuously and boil for 15 min so that the precipitate is properly formed. Allow the solution to stand at just below boiling point for at least 30 min, check for complete precipitation by adding 2 to 3 drops of barium chloride solution, and leave to cool for 12 h to 24 h. Filter the precipitate on a fine filter paper¹³⁾ and wash until free of chlorides with hot distilled water. Place the paper and its contents in a weighed crucible and ignite to constant mass at 800 °C to 900 °C. Some reduction of the barium sulphate may occur during ignition but by adding 2 to 3 drops of sulphuric acid ($d = 1.84$) and re-igniting re-oxidation will occur.

$$\% \text{ SO}_3 = \frac{\text{precipitate mass (as BaSO}_4) \times 34.3 \times 5}{\text{sample mass}}$$

NOTE If the silica precipitate has been treated with hydrofluoric acid and the residue fused and added to the stock test solution, the latter cannot be used for this determination. A new solution of the sample should be prepared as in 6.3.2.3 but with the procedure in note 2 omitted.

6.3.2.6 Total iron (as Fe₂O₃). Transfer a 5 ml aliquot of the "stock test solution" to a 100 ml graduated flask, add 2 ml of hydroxylammonium chloride solution (100 g/litre) and 5 ml of freshly prepared 1,10-phenanthroline solution (10 g/litre). Add successive 2 ml portions of ammonium acetate solution (approximately 100 g/litre) until the red colour of the complex appears, followed by a further 2 ml.

Allow to stand for 15 min, dilute to 100 ml and mix. Measure the optical density of the solution against water in 10 mm cells at 510 nm or by using a colour filter¹⁴⁾ in a suitable instrument. The colour is stable for between 15 min and 75 min after the addition of the ammonium acetate solution.

Determine the ferric oxide content of the solution by reference to a calibration graph (6.2).

$$\% \text{ Fe}_2\text{O}_3 = \frac{\text{mg (Fe}_2\text{O}_3) \text{ in solution} \times 10}{\text{sample mass}}$$

¹²⁾ No. 40 Whatman or similar filter paper has been found to be suitable.

¹³⁾ No. 42 Whatman or similar filter paper has been found to be suitable.

¹⁴⁾ An Ilford 603 filter has been found to be suitable.