

Mortar — Methods of test for mortar — Chemical analysis and physical testing

ICS 91.100.10

Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Subcommittee B/519/2, Mortar, upon which the following bodies were represented:

Autoclaved Aerated Concrete Products Association

Brick Development Association

British Cement Association

British Lime Association

CERAM

Cementitious Slag Makers Association

Civil Testing Machine Manufacturers' Association

Department of the Environment (Building Research Establishment)

Federation of Plastering and Drywall Contractors

Institute of Concrete Technology

Institution of Structural Engineers

National House-Building Council

National Specialist Contractors Council

Quarry Products Association

United Kingdom Quality Ash Association

Vermiculite Association

Co-opted members

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 22 August 2005

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First published as BS 4551-1 and BS 4551-2 February 1998

The following BSI references relate to the work on this British Standard:
Committee reference B/519/2
Draft for comment 05/30125494

Amendments issued since publication

Amd. No.	Date	Comments

ISBN 0 580 46454 7

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Foreword

This British Standard has been prepared by Subcommittee B/519/2. It supersedes BS 4551-2:1998, and partially supersedes BS 4551-1:1998, which are withdrawn. The remainder of BS 4551-1:1998 has already been superseded by BS EN 1015-1:1999, BS EN 1015-2:1999, BS EN 1015-3:1999, BS EN 1015-4:1999, BS EN 1015-6:1999, BS EN 1015-7:1999, BS EN 1015-9:1999, BS EN 1015-10:1999, BS EN 1015-11:1999 and BS EN 1015-19:1999.

The revision specifies the chemical analysis of mortar not covered in the BS EN 1015 series, which was formerly in BS 4551-2:1998. It also includes a physical test method for water retentivity and a physical test method for the determination of consistence by dropping ball. This was formerly in BS 4551-1:1998.

The requirements in this standard cover areas that are not addressed by BS EN 998-1, BS EN 998-2 and BS EN 1015 (all parts).

This British Standard describes methods of testing for mortar only, and should not be used or quoted as a specification for mortar. References to this standard should indicate that the methods of testing used are in accordance with BS 4551.

Only general guidance is given here, because of the variety of circumstances in which sampling of hardened mortars might be required.

Samples might 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 or rendering.

For this purpose, it should be realised that a number of different batches might have been used during construction, and a number of different 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 this should be considered when interpreting and reporting 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.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its 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 37 and a back cover.

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1 Scope

This British Standard specifies methods of sampling preparation, physical testing and chemical analysis of mortars for bricklaying, plastering and rendering. The methods described are for the determination of parameters that have traditionally been used in the UK but which are neither defined, nor superseded, by BS EN 998-1, BS EN 998-2 or BS EN 1015 (all parts).

Methods of interpretation of chemical analysis results are also described.

The methods of test given in this standard are not intended to be applied to mortars containing high alumina cement or mortars whose principle binder is hydraulic lime.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced documents (including any amendments) applies.

BS 410, *Test sieves — Technical requirements and testing*.

BS 5404-1, *Specification for plastic laboratory ware — Part 1: Beakers*.

BS EN 933, *Tests for geometrical properties of aggregates — Determination of particle size distribution*.

BS EN 1015-2, *Methods of test for mortar for masonry — Part 2: Bulk sampling of mortars and preparation of test mortar*.

BS EN 6100-6.6.1, *Glossary of building and civil engineering terms — Part 6: Concrete and plaster — Section 6.6: Products, applications and operations — Subsection 6.6.1: Concrete and mortar*.

BS EN ISO 3696, *Water for analytical laboratory use — Specification and test methods*.

3 Terms and definitions

For the purposes of this British Standard, the terms and definitions given in BS 6100-6.6.1 and the following apply.

3.1 admixture

material added in small quantities to produce specified modifications to the properties

3.2 aggregate

granular material that does not contribute to the hardening reaction of the mortar

3.3 binder

material used to hold solid particles together in a coherent mass, e.g. cement, building lime

3.4 lime

material comprising any physical and chemical forms under which calcium and/or magnesium oxide (CaO and/or MgO) and/or hydroxide (Ca(OH)₂ and/or Mg(OH)₂) can appear

3.5 hydrated limes

air limes, calcium limes or dolomitic limes, resulting from the controlled slaking of quicklimes (they are produced in the form of a dry powder or putty or as a slurry (milk of lime))

3.6 masonry mortar

mix of one or more inorganic binders, aggregates, water, and sometimes additions and/or admixtures for bedding, jointing and pointing of masonry

3.7 render/plaster

materials used externally are referred to as render/rendering and materials used internally as plaster/plastering

3.8 rendering mortar

mix of one or more inorganic binders, aggregates, water and sometimes admixtures and/or additions, used as external renders or internal plasters

NOTE Rendering mortar is also known as plastering mortar.

4 Symbols and designations

For the purposes of this British Standard, the following symbols and designations apply.

<i>A</i>	insoluble residue (obtained by analysis)
<i>a</i>	content of free water as a percentage of the wet mass of the mortar
<i>B</i>	percentage soluble SiO ₂
<i>b</i>	loss in mass (g)
<i>C</i>	percentage soluble SiO ₂ (or CaO) to be subtracted
<i>c</i>	sample mass (g)
<i>D</i>	percentage of free water by wet mass
<i>d</i>	percentage of available lime as Ca(OH) ₂ by wet mass
<i>e</i>	percentage of available lime as C(OH) ₂ by dry mass
<i>F</i>	percentage soluble CaO
<i>f</i>	percentage of available lime by wet mass
<i>G</i>	percentage soluble CaO to be subtracted
<i>g</i>	total lime content as the percentage of CA(OH) ₂ by wet mass
<i>H</i>	the average result
<i>h</i>	the content of (lime + silt + clay) as a percentage of dry mass
<i>I</i>	percentage of silt and clay by dry mass
<i>i, ii, iii, iv, v</i>	traditional mortar designations (see 9.3.1)
<i>J</i>	content of fine aggregate (sand) as a percentage of dry mass
<i>j</i>	percentage of lime, silt and clay
<i>K</i>	molarity of ammonium thiocyanate (NH ₄ SCN) solution
<i>k</i>	percentage of insoluble residue in the sample
<i>L</i>	percentage SO ₃
<i>l</i>	percentage of soluble silica in the sample
<i>M</i>	quantity of wet sample, equivalent to 100 g dried mortar
<i>m</i>	mass of ignited residue
<i>N</i>	relative density of liquid reagent
<i>n</i>	percentage of calcium oxide in the sample
<i>o</i>	percentage of sulphur trioxide in the sample
<i>p</i>	precipitate mass (as BaSO ₄)
<i>q</i>	percentage of total iron in the sample
<i>R</i>	total mass (mg) of dried residues on the test sieves
<i>R</i> ₁	reproducibility conditions
<i>R</i> ₂	reproducibility limit (see B.2.4)
<i>r</i>	mg Fe ₂ O ₃ in solution
<i>r</i> ₁	is the repeatability limit (see B.2.4)
<i>s</i>	CaO equivalent of EDTA (mg)
<i>T</i>	titre of EDTA

t	percentage of aluminium oxide in the sample
u	percentage of magnesium oxide in the sample
V	variance
V_1	volume of 0.1 M silver nitrate (AgNO_3)
V_2	titre of ammonium thiocyanate ($\text{NH}_4 \text{CSN}$) solution
V_3	volume of EDTA standard solution used for the calcium and magnesium titration (ml)
V_4	volume of EDTA standard solution used in the calcium titration (ml)
V_5	volume of the HCl solution required (ml)
V_6	volume of DCTA standard solution (0.05 M) (ml)
V_7	volume of standard zinc solution (0.05 M) used in the back-titration (ml)
w	MgO equivalent (mg)
X	molarity of the HCl solution
y	percentage of chloride in the sample
z	percentage aggregate in mortar

5 Sampling

5.1 Principle

The mortars are sampled and reduced on-site to a quantity suitable for dispatch to a laboratory, in order to conduct physical tests and chemical analysis. As part of this process, they are packaged and labelled.

5.2 Freshly mixed mortars

Fresh mortar shall be sampled, reduced, packaged and certificated in accordance with BS EN 1015-2.

The method to be used to bring test samples to the required consistence shall be specified when preparing them from dry constituents. All deviations from the requirements in BS EN 1015-2 shall be noted on the certificate and included in the report (see 6.1.5).

NOTE If consistence by dropping ball and air content tests are to be determined, arrangements should be made to carry out the tests at the point of sampling.

Before testing, the sample shall be gently stirred by hand using a trowel or palette knife in 5 s to 10 s, but without any additional mixing of the batch.

5.3 Hardened mortars

5.3.1 Apparatus

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

5.3.1.2 *Means of collecting representative samples*, without loss of fine material.

5.3.1.3 *Containers*, which will prevent sample loss or contamination.

5.3.2 Size of samples

Samples for dispatch 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, 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
Masonry mortar	50	50
Plastering or rendering mortar	50	50

5.3.3 Taking of samples and source

5.3.3.1 Masonry mortar

Take the corresponding mass of sub-sample, as specified in Table 1, to produce a representative quantity of mortar by removing a number of bricks, blocks or other units from the construction. For most purposes, carefully remove the mortar from the substrate, avoiding contamination with the material of which the bricks or blocks are composed. Take each sub-sample across 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 masonry units, 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 masonry units are composed, and that there is no loss of the finer material.

5.3.3.2 Plastering and rendering mortar

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

5.3.4 Packaging and certificate of sampling

Each sample to be dispatched to a laboratory shall be placed in one or more suitably labelled containers 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 is relevant:

- name and address of the body responsible for sampling;
- name and address of the customer;
- place, date, time and method of producing the test sample;
- location in the building of the area sampled, and the depths from the face of the structure represented by the sample;
- state of the mortar at the time of sampling, i.e. wet or dry, soft or hard;
- available information 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 the investigation;
- specified mix, origin and age of the sample, if known;
- identification mark on the sample container;
- remarks.

6 Physical tests

6.1 Determination of consistence by dropping ball

6.1.1 Principle

The dropping ball penetration is determined either at the point of sampling for site-made mortars or in the laboratory for mixes made there and reported to determine the consistence.

6.1.2 General

When testing site-made materials, the dropping ball penetration shall be determined at the point of sampling and reported. For physical tests on mortar mixes prepared in the laboratory, the consistence shall be adjusted to a penetration of (10 ± 0.5) mm.

Preliminary tests in accordance with this clause shall be undertaken in order to determine the appropriate water content.

6.1.3 Apparatus

6.1.3.1 Mould made from a rigid material, 100 mm internal diameter, 25 mm internal depth.

6.1.3.2 Palette knife.

6.1.3.3 Methyl methacrylate ball, having a diameter of (25 ± 0.1) mm, and a mass of (9.8 ± 0.25) g with its surface polished.

6.1.3.4 Dropping mechanism for the ball, that does not impart to the ball any appreciable spin, friction or acceleration, other than that due to gravity, in the process of release, and ensures that the ball falls freely and drops vertically within the limits specified in **6.1.4**. A suitable apparatus is shown in Figure 1.

6.1.3.5 Device for measuring the depth of penetration of the ball, in millimetres, with an accuracy of 0.1 mm, without imposing any pressure on the ball in the process of measurement. The device shall also be suitable for measuring any fall in the level of the surface of the mortar in the test for consistence retentivity (see **6.2**).

NOTE 1 A suitable measuring device is shown in Figure 2.

NOTE 2 In both tests the mortar is initially trowelled flat and level with the top of the mould, and this level is used as the datum for measurement. Instruments with a tripod stand that rests on the rim of the mould are therefore suitable. Such tripod stands should allow for sufficient sliding movement to permit measurement of the penetration of a ball in any position within the 12 mm from the centre of the surface that is allowed in **6.1.4**.

NOTE 3 The requirement for no pressure to be imposed on the ball in the process of measurement is most easily satisfied by a device that incorporates a measuring foot, having a horizontal base of sufficient width to ensure that measurements are made on the highest point of the ball, and that can be brought slowly down on the ball whilst sighting across the top of the ball. A white sight screen behind the ball helps to determine when the measuring foot just touches the ball.

6.1.4 Procedure

When testing mortar prepared in the laboratory from dry constituents, test the mortar within (150 ± 15) s of the completion of mixing. Otherwise, record the age of the mortar.

Fill the mould with the mortar, in about 10 increments, by pushing the mortar into it with the end of a palette knife. When the mortar is slightly above the rim of the mould, strike off the surface plane and level with the top of the mould, using a palette knife. Hold the knife at about 45° and move it across the mould once, with a sawing motion, to strike off excess material, and then at a slightly flatter angle to trowel the surface in a single action in the reverse direction. The surface shall not be trowelled further unless the consistence of the mortar is so low that the two movements are insufficient to produce the required finish.

Place the apparatus on a flat, horizontal surface free from vibration. The methyl methacrylate ball shall be allowed to fall freely through a height of 250 mm before striking the surface of the mortar to land within 12 mm of the centre of the surface. Measure the penetration of the ball and record it to the nearest 0.1 mm. Remove the ball from the mortar, rinse, wipe with a clean cloth and replace in the release mechanism; return the mortar to the mixing bowl (unless it is to be used for the consistence retentivity or water retentivity tests).

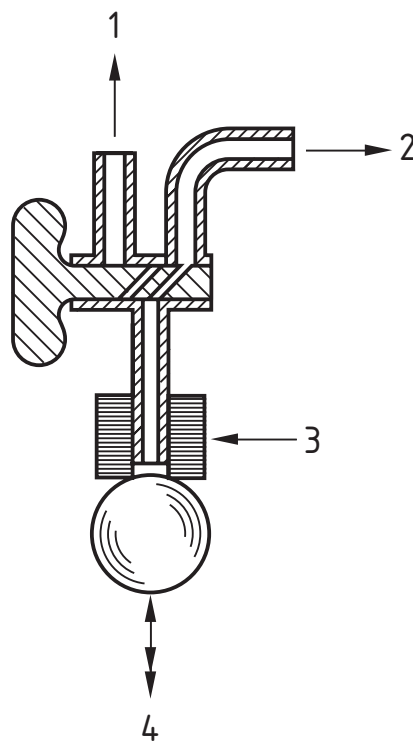
Three tests shall be conducted on separate portions of the mortar.

NOTE The tests for consistence retentivity and water retentivity may be done in conjunction with this test on the same specimen.

6.1.5 Test report

The test report shall include the following:

- a) the average of the three penetrations, to the nearest 0.1 mm, as the consistency, calculated as specified in 6.1.4;
- b) all information necessary for identification of the sample tested;
- c) a reference to this British Standard, i.e. BS 4551:2005;
- d) any deviations from the procedure specified;
- e) any unusual features (anomalies) observed during the test;
- f) the date of the test.



Key

- 1 open air
- 2 to suction source
- 3 rubber bung
- 4 250 mm above surface of mortar

Figure 1 — Diagrammatic representation of ball release

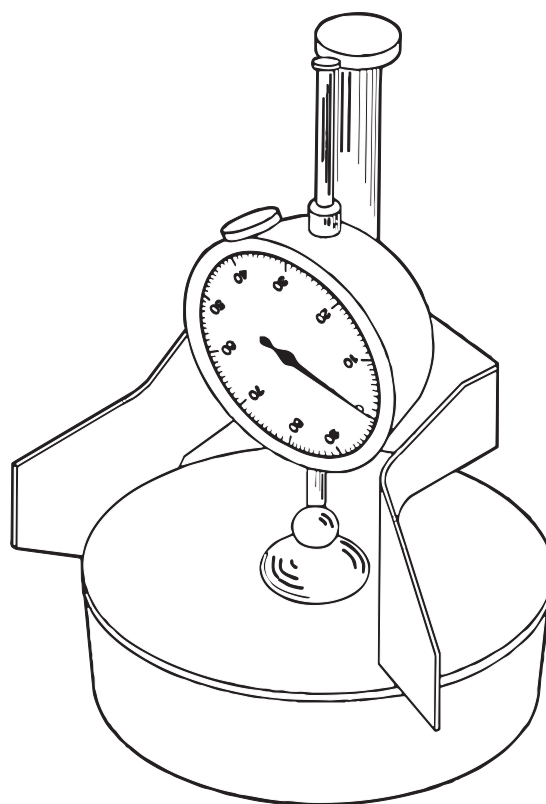


Figure 2 — Suitable device for measuring penetration

6.2 Determination of consistence retentivity and water retentivity

NOTE 1 The retention of consistence and of water in mortars is of practical importance, particularly if the mortar is to be applied on materials of high suction. Since the consistence of a given mortar is dependent upon both water content and entrained air content, it is not sufficient with all mortars to measure the water retained under a standard test condition. The degree to which the mortar retains its consistence may be the more useful measure for general use.

NOTE 2 This clause deals with two tests to determine the properties of the mortar before and after applying a standard suction treatment to the mortar.

In one test the consistence is measured and the final consistence value, expressed as a percentage of the original value, is termed the “consistence retentivity”.

In the other test, the mass of the water retained after suction is measured and, when expressed as a percentage of the original water content, is termed the “water retentivity”. It is of value in comparing mortars of closely similar type.

6.2.1 Principle

In this procedure, a metal mould is filled with mortar in a prescribed manner and a methyl methacrylate ball of known dimensions and mass is then dropped onto the mortar from a prescribed height. The penetration is then measured and reported as the consistence.

6.2.2 Apparatus

6.2.2.1 *Mould made from a rigid material, as described in 6.1.3.1.*

6.2.2.2 *Palette knife.*

6.2.2.3 *A 2 kg weight.*

6.2.2.4 *Rigid non-porous plate, 110 mm in diameter.*

6.2.2.5 *Two discs 110 mm in diameter or two squares of 100 mm side, of white cotton gauze. The discs shall be 110 mm in diameter and the squares shall be of side 100 mm.*

6.2.2.6 *Eight discs of extra white filter paper, 200 g/m², 110 mm in diameter.*

6.2.3 Procedure

Either test the mortar within (150 ± 15) s of the completion of mixing when testing mortar prepared in the laboratory from dry constituents, or record the age of the mortar.

NOTE These tests may be done in conjunction with the dropping ball method for measuring consistence (see 6.1), but the mortar used should normally be discarded after completion of these tests. Both of these measurements can be made on the same filling of the mould.

Weigh the mould in a dry condition and weigh eight discs of filter paper. Fill the mould and strike off plane and level in two movements only, as described in 6.1.4. Remove all the mortar from the outside of the mould and determine the average penetration as described in 6.1.4. Fill the depression left by the ball with mortar, strike off plane and level in two movements as described in 6.1.4. Weigh the mould and its contents.

Either cover the surface of the mortar with the two pieces of cotton gauze, and place the eight discs of filter paper on top of the gauze. Place the non-porous plate on top of this and load with the 2 kg mass. After 2 min, remove the mass, discard the cotton gauze and weigh the filter papers to the nearest 0.05 g.

Alternatively, use the mass of the full mould to define the water removed, in which case it is unnecessary to first weigh the filter paper.

Calculate the mass of water originally present in the mould from the mass of mortar in the mould and the moisture content of the mortar. Where the moisture content is not known from the composition of the mortar, determine it at the same time by the method described in 7.5.2.1. The mass of water retained by the mortar after suction (i.e. the mass of water originally present in the mould minus the mass of water absorbed by the filter paper), expressed as a percentage of the mass of water originally present in the mould full of mortar, shall be taken as the water retentivity.

Measure any fall in level of the mortar. Make a single measurement, by the dropping ball method, on the mortar after suction. Correct the apparent penetration of the ball by subtracting from it any measured fall in the level of the mortar. The corrected penetration of the ball after suction, expressed as a percentage of the average penetration before suction, shall be calculated as the consistence retentivity.

Repeat this procedure with a second sample of mortar.

6.2.4 Test Report

The test report shall include the following:

- a) the average of the two water retentivity values to the nearest 1 %, calculated as specified in 6.2.3;
- b) the average of the two consistence retentivity values to the nearest 5 %, calculated as specified in 6.2.3;
- c) all information necessary for identification of the sample tested;
- d) a reference to this British Standard, i.e. BS 4551:2005;
- e) any deviations from the procedure specified;
- f) any unusual features (anomalies) observed during the testing;
- g) the date of the test.

7 Chemical analysis

7.1 Principle

Chemical analysis is performed on a sample of mortar in order to estimate the original mix proportions.

7.2 Apparatus for chemical analysis

NOTE 1 It is assumed that a laboratory carrying out these tests is 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.

7.2.1 *Volumetric glassware, with an accuracy of class B or better (where given) in BS 700-1.*

7.2.2 *250 ml polypropylene beakers, conforming to BS 5404-1.*

7.2.3 *Furnace, or furnaces, controlled to maintain temperatures of (850 ± 25) °C, (925 ± 25) °C and $(1\ 200 \pm 25)$ °C.*

7.2.4 *Boiling water or steam bath.*

7.2.5 *Test sieves*, conforming to BS 410.

7.2.6 *Pestle and mortar, or mechanical crusher.*

7.3 Reagents for chemical analysis

7.3.1 Unless otherwise stated, all reagents shall be of analytical quality. Distilled or de-ionised water conforming to BS EN ISO 3696 shall be used throughout the analysis. Reagents shall be those provided in **7.3.2** to **7.3.48**. Where reagents are prepared in the laboratory, the procedures given in **7.4** 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). This 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 might cause spurting.

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

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

7.3.2 *Acetic acid*, glacial.

7.3.3 *Ammonia solution*, *N* = 0.880.

7.3.4 *Ammonium acetate buffer*. Add, with stirring, 120 ml of acetic acid (see **7.3.2**) to 500 ml of water, followed by 74 ml of ammonia solution (see **7.3.3**). Cool, dilute to 1 l and mix.

7.3.5 *Ammonium acetate solution*, 100 g/l. Dilute 70 ml of acetic acid (see **7.3.2**) to 1 l with water and add carefully 70 ml of ammonia solution (see **7.3.3**). Mix, cool and adjust to pH 6, either with acetic acid or ammonia solution.

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

7.3.7 *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.

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

7.3.9 *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.

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

7.3.11 *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 part hydrochloric acid + 4 parts of water). Boil to expel carbon dioxide, cool and dilute to 1 l in a volumetric flask.

7.3.12 *Chloroform*.

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

7.3.14 *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 **7.3.18**). 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 **7.3.17**) 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 **7.3.48**) from blue-green to the first appearance of a permanent pink colour.

7.3.15 Dithizone indicator solution. Dissolve 0.012 g of dithizone in 50 ml of ethanol (see 7.3.17).

NOTE This solution will keep for about one week.

7.3.16 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 magnesium standard solution (see 7.3.28) into a 500 ml conical flask. Add 10 ml of potassium hydroxide solution (see 7.3.38) and dilute to about 200 ml. Add about 0.015 g of screened calcein indicator and titrate with the EDTA standard solution (see 7.3.16) 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 7.3.28) into a 500 ml conical flask. Add 20 drops of hydrochloric acid (see 7.3.18), 20 ml of ammonia solution (see 7.3.3) and dilute to about 200 ml. Add 0.04 g of methylthymol blue complexone indicator and titrate with the EDTA standard solution (see 7.3.16) from blue to colourless. Calculate the MgO equivalent of the EDTA standard solution in milligrams of MgO per ml.

7.3.17 Ethanol, 95 %. Industrial methylated spirit.

NOTE Ethanol may be replaced for this purpose by industrial methylated spirits, 66° O.P., conforming to BS 3591. Attention is drawn to the fact that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1952 (SI 1952, No. 2230) [1].

7.3.18 Hydrochloric acid, $N = 1.18$.

7.3.19 Dilute hydrochloric acid, (1 part dilute hydrochloric acid + 9 parts of water).

7.3.20 Dilute hydrochloric acid, (1 part dilute hydrochloric acid + 49 parts of water).

7.3.21 Hydrochloric acid, approximately 0.5 M. Dilute 45 ml of hydrochloric acid (see 7.3.18) to 1 l. Standardize against sodium carbonate standard solution (see 7.3.41) using methyl orange as the indicator.

7.3.22 Hydrofluoric acid, $N = +1.13$ or 40 % (m/m).

7.3.23 Hydrogen peroxide, 6 %.

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

7.3.25 Iron III indicator solution. Add 10 ml of nitric acid (see 7.3.32) to 100 ml of cold saturated solution of ammonium ferric sulfate.

7.3.26 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 7.3.23). Boil for 15 min to decompose the excess of hydrogen peroxide. Cool and dilute to 1 l in a volumetric flask.

7.3.27 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. To each, add 2 ml of hydroxylammonium chloride solution (see 7.3.24), 5 ml of 1,10-phenanthroline solution (see 7.3.33) and 2 ml of ammonium acetate solution (see 7.3.5). 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 filter¹⁾ has been found to be suitable.

¹⁾ Ilford 603 filter is a trade mark owned by Ilford Imaging UK Limited, Town Lane, Mobberley, Knutsford, Cheshire WA16 7JL, United Kingdom and is an example of a suitable product available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by BSI of this product..

7.3.28 Magnesium standard solution, 0.1 mg MgO/ml. Wash the metal in hydrochloric acid (see 7.3.19) 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 7.3.19), cool and dilute to 1 l in a volumetric flask.

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

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

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

7.3.32 Nitric acid, $N = 1.42$.

7.3.33 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 part acetic acid + 1 part water). (See 7.3.2.)

7.3.34 Phenolphthalein indicator solution. Dissolve 1 g of phenolphthalein in 100 ml of ethanol. (See 7.3.17.)

7.3.35 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.

7.3.36 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. Stir, preferably using a mechanical stirrer, until dissolved. Discard after two weeks.

7.3.37 Potassium hydrogen sulfate, solid.

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

7.3.39 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.

Or, dissolve 10.788 g of pure silver metal in the minimum amount of nitric acid (see 7.3.32) and dilute to 1 l.

7.3.40 Sodium carbonate solution, 100 g/l. Dissolve 100 g of anhydrous sodium carbonate, dried at 150 °C, in water and dilute to 1 l in a volumetric flask.

7.3.41 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.

7.3.42 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 dilute to 1 l. Standardize against the hydrochloric acid solution (see 7.3.21) using phenolphthalein as indicator.

7.3.43 Sugar, granulated.

7.3.44 Sulfuric acid, $N = 1.84$.

7.3.45 Triethanolamine, (1 part triethanolamine + 1 part water).

7.3.46 Triethanolamine, (1 part triethanolamine + 4 parts water).

7.3.47 Trimethyl hexanol (nonyl alcohol).

7.3.48 Standard zinc solution, 0.05 M. Wash the metal in hydrochloric acid (see 7.3.19) to remove any oxide film. Then wash 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 7.3.18) 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.

7.4 Procedure for laboratory preparation of samples for chemical analysis

7.4.1 General

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 conform to BS 410.

7.4.2 Freshly mixed lime: fine aggregate (sand) for mortars

Thoroughly mix each sample and reduce to about 250 g. Place in a wide-mouthed jar and enclose with an airtight cap. Immediately before starting each of the tests in 7.5.1, remix the reduced sample with a spatula.

7.4.3 Freshly mixed cement-based mortars

NOTE By the time the sample has arrived in the laboratory, hardening has normally occurred.

Dry the sample immediately on receipt at $(105 \pm 5) ^\circ\text{C}$ 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. Reduce to 250 g.

Treat the sample in accordance with 7.4.5.4 and 7.4.5.5.

7.4.4 Freshly mixed gypsum-based plasters

As gypsum-based backing plasters set reasonably quickly, the sampling shall follow the method described in 7.4.5.3 to 7.4.5.5.

7.4.5 Hardened mortars and plasters

NOTE The preparation often involves separating, by suitable means, various layers of a multilayer 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.

7.4.5.1 Where there is difficulty in finding the interface between what are reputedly two layers of mortar or plaster, take samples as near to the outside faces of the composite layer as is possible, or, if the composite layer is too thin to allow this, analyse it as one layer, making an appropriate note to that 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.

7.4.5.2 Measure the mortar or plaster sample to determine the average thickness. Examine the uniformity of samples to establish efficiency of mixing, and note the presence of nodules of fine aggregate (sand), etc. Report all of this information with the other tests on the sample.

7.4.5.3 Crush the sample to pass a 10 mm test sieve, avoiding as far as possible the fracturing of aggregate particles, and reduce to 250 g. Dry this sample at $(105 \pm 5) ^\circ\text{C}$ in the case of cement-based materials, or at $(40 \pm 2) ^\circ\text{C}$ for gypsum-based materials, to constant mass. Place in a jar and enclose using an airtight cap.

7.4.5.4 When aggregate grading is required, sufficient test samples, each of about 50 g mass, shall be taken by an efficient sampling method. (See Clause 5.)

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

7.4.5.6 If suitable mechanical grinding equipment is not available, 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. 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 commences.

7.5 Procedures**7.5.1 General**

NOTE 1 This clause describes a method for determining the grading of the aggregate used in the mix, but it should be stressed that, where aggregate contains acid soluble material, the result will be inaccurate. It is also possible that, as test methods differ from those given in BS 812-120:1989, slightly different results may be obtained.

Soluble silica shall be extracted according to the method given in 7.5.3.

Methods of proven accuracy are given for the determination of soluble silica and other compounds in the mortar. Alternative methods may be used for the following compounds, provided evidence is presented that such methods give equivalent results:

- a) calcium oxide (see 7.5.3.4), e.g. by atomic absorption spectroscopy;
- b) soluble silica (see 7.5.3.3), e.g. by atomic absorption spectroscopy;
- c) total iron (see 7.5.3.6), e.g. by atomic absorption;
- d) aluminium oxide (see 7.5.3.7), e.g. by atomic absorption spectroscopy;
- e) magnesium oxide (see 7.5.3.8), e.g. by atomic absorption spectroscopy;
- f) chloride (see 7.5.3.9), by potentiometric titration.

NOTE 2 Duplicate analyses should normally be made, but a single analysis might suffice when a series of samples from a single source is being analysed.

NOTE 3 Where mix proportions only are required, the analysis need not be continued beyond the method described in 7.5.3.4 or, in the case of gypsum-based materials, the method in 7.5.3.5.

7.5.2 Ready-mixed lime: fine aggregate (sand) for mortars

NOTE 1 In this clause, the methods apply only to mortars consisting of lime and lime aggregate. Attention is drawn to the practical limitation of the method where such mortars 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 7.5.2.2 gives only a minimum value for the available lime content. This value should be supplemented by a determination of the total lime content (see 7.5.2.3) to the maximum amount of lime that could have been present. It should be recognized that this maximum value includes any calcium compounds that might have been present in the lime aggregate (sand) used.

NOTE 2 If the analysis of the fine aggregate (sand) is known, a correction can be applied to the total lime content and the actual available lime content determined. If this is done, it will not be necessary to determine the available lime content by the method given in 7.5.2.2.

7.5.2.1 Free water content

Weigh (10 ± 0.1) g of the sample into a dry pre-weighed dish and dry at $(105^\circ \pm 5)^\circ\text{C}$ to constant mass.

The content of free water as a percentage of the wet mass of the mortar to the nearest 0.5 %, a , is given by the following equation:

$$a = \frac{b}{c} \times 100$$

where

- b is the loss in mass (g);
- c is the sample mass (g).

NOTE Sample drying times should be minimized and samples should be sealed in a container immediately after testing to minimize carbonation.

7.5.2.2 Available lime content

Weigh (5 ± 0.05) g of the sample (see 7.4.2) into a 250 ml flask containing 20 ml to 30 ml of water. Immediately stopper the flask loosely with a rubber stopper. Disperse the sample thoroughly in the water with 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 paper. The filtrate should be collected in a Buchner flask and titrated immediately to avoid carbonation.

Add five drops of phenolphthalein indicator solution and wash down the stopper and sides of the flask with water. 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 as a percentage of calcium hydroxide ($\text{Ca}(\text{OH})_2$) by wet mass and as a percentage of $\text{Ca}(\text{OH})_2$ by dry mass to the nearest 0.1 %, from the following equations.

The percentage of available lime as $\text{Ca}(\text{OH})_2$ by wet mass, d , is given by the equation:

$$d = \frac{3.7 V_5 X}{c}$$

where

- V_5 is the volume of the HCl solution required (ml);
- X is the molarity of the HCl solution;
- c is the sample mass (g).

The percentage of available lime as $\text{Ca}(\text{OH})_2$ by dry mass, e , is given by the equation:

$$e = \frac{f}{100 - D} \times 100$$

where

- f is the percentage of available lime by wet mass;
- D is the percentage of free water by wet mass.

NOTE This test method might underestimate the lime content of a mortar at the time of manufacture if the test sample has subsequently undergone carbonation. The potential for this effect should be included in the test report.

7.5.2.3 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 (see 7.3.18) and mix thoroughly. Add about 100 ml water, heat to boiling and simmer for 5 min. Filter through a coarse, hardened filter paper (Whatman No. 541²⁾ or equivalent) 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 10 ml to a 500 ml conical flask using a pipette. Add 5 ml triethanolamine solution (1 part triethanolamine + 1 part water) and 10 ml potassium hydroxide solution (see 7.3.38) and dilute with approximately 200 ml of water. Add about 0.15 g screened calcein indicator (fluorescein complexone) and, while stirring, titrate against a black background using EDTA standard solution (see 7.3.16) until the colour changes from fluorescent green to pink-purple.

Calculate the total lime content as the percentage of $\text{Ca}(\text{OH})_2$ by wet mass to the nearest 0.1 %, g , from the following formula:

$$g = T \times \frac{74.1}{56.08} \times \frac{5s}{c}$$

where

- s is the CaO equivalent of EDTA (mg);
- c is the sample mass (g);
- T is the titre of EDTA.

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

7.5.2.4 Fine aggregate (sand), silt and clay content and aggregate grading

Ensure the sieves have been wetted on both sides. Arrange with the coarsest sieve on top and the finest sieve at the bottom. Weigh a quantity, M , of the wet sample (see 7.4.2), equivalent to 100 g dried mortar and calculated to the nearest 0.1 g, from the formula:

$$M = \frac{100 \times 100}{100 - D}$$

where

- D = percentage of free water by wet mass.

²⁾ Whatman No. 541 is a trade mark owned by Whatman plc, 27 Great West Road, Brentford, Middlesex TW8 9BW, United Kingdom and is an example of a suitable product available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by BSI of this product.

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, 2.35 mm, 1.18 mm, 600 μm , 300 μm , 150 μm and 75 μm .

Agitate in a sufficiently vigorous way, 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 to be removed by decantation of the wash water. Repeat until the wash water is clear.

NOTE 1 Take care to avoid, as far as possible, decantation of the coarser particles of the sample.

Transfer all the material in the container 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 $(105 \pm 5)^\circ\text{C}$. After cooling to room temperature, brush the residue from each sieve and weigh each residue separately to obtain the fine aggregate (sand) grading. Calculate the sieve analysis of the fine aggregate (sand) to determine the aggregate grading as described in BS 812.

Calculate the content of (lime + silt + clay) by dry mass to the nearest 0.1 %, h , from the following formula:

$$h = 100 - R$$

where

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

Using the available lime content of the mortar (as determined in 7.5.2.2, or in 7.5.2.3 if the analysis of the fine aggregate (sand) is known), calculate the respective contents of lime, (silt + clay), and fine aggregate (sand) as a percentage of dry mass to the nearest 0.1 %.

The percentage of lime by dry mass is the percentage of available lime, as a percentage of $\text{Ca}(\text{OH})_2$ by dry mass.

The content of silt and clay as a percentage of dry mass to the nearest 0.1 %, I , is calculated from the following formula:

$$I = j - e$$

where

j is the percentage of lime, silt and clay;

e is the percentage lime as a percentage $\text{Ca}(\text{OH})_2$ by dry mass.

The content of fine aggregate (sand) as a percentage of dry mass to the nearest 0.1 %, J , is calculated from the following formula:

$$J = 100 - e$$

where

e is the percentage lime as $\text{Ca}(\text{OH})_2$ by dry mass.

NOTE It is assumed that the lime used in the mortar consists of pure $\text{Ca}(\text{OH})_2$ in the dry state and that it has passed the 75 μm test sieve. It is also assumed that the aggregate is washed completely clean of lime. These assumptions are unlikely to be correct. It is therefore necessary to make appropriate corrections where the relevant data are known, to take this into account when interpreting results and to note this in the test report.

7.5.3 Freshly mixed and hardened mortars and plasters

7.5.3.1 General

Follow the described method for the extraction of soluble silica that gives methods for the determination of soluble silica and other oxides in the extract.

Make samples of the raw materials available for freshly mixed materials, if possible. The fine aggregate (sand) and cement, or gypsum, shall be analysed by the same method as the mortar or plaster that they constitute.

Where the constituents of hardened materials are available, they shall also be analysed.

NOTE 1 Particular care should be taken to ensure that the extraction and measurement of soluble silica are identical for both mortar and fine aggregate (sand) and cement or gypsum.

NOTE 2 Building lime should be analysed by the methods described in BS EN 459 and BS 6463.

NOTE 3 When samples of the constituents are not available, the analysis of the mortar might 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 7.5.3.4) having a volume of 500 ml and on the use of the specified aliquots. The formulae might need modification where alternative methods are used.

7.5.3.2 Fine aggregate (sand), silt and clay content, aggregate grading

Transfer the 50 g sample (see 7.4.5), accurately weighed, to a 1 l beaker. Add 500 ml hydrochloric acid (see 7.3.19) 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 (see 7.3.36), allow to settle and decant the liquid through a pre-weighed, hardened, ashless filter paper. Wash the fine aggregate (sand) remaining in the beaker by decantation three times with about 200 ml of 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 (see 7.3.40). Simmer gently for between 15 min and 20 min, taking care to avoid loss by frothing. Transfer the contents, through a small 75 µm test sieve conforming to BS 410, into the original paper. Wash six times with ammonium chloride solution (see 7.3.6), twice with hydrochloric acid (see 7.3.19) and twice with hot water. Dry the sieve residue at (105 ± 5) °C. Add the material remaining on the filter paper, weigh and report this as silt and clay content.

Determine the aggregate grading in accordance with BS EN 933.

NOTE Where the aggregates contain acid-soluble material, the fine aggregate (sand) grading test is inaccurate.

7.5.3.3 Insoluble residue and soluble silica

Except where low cement contents are expected, weigh (5 ± 0.005) g of the analytical sample, prepared as specified in 5.3, into a 250 ml polypropylene beaker. Carefully add 100 ml of hydrochloric acid (see 7.3.19). 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 pre-weighed, hardened, ashless paper pre-dried at (105 ± 5) °C, cooled and stored in a desiccator.

NOTE 1 When low cement contents are expected, a sample mass of (10 ± 0.005) g can be taken at the analyst's discretion.

NOTE 2 The rate of filtration is improved by adding 2 ml of polyacrylamide solution (see 7.3.35) to the acid extraction just before 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 (see 7.3.20), 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 (see 7.3.40). Place the beaker on a boiling water or steam bath and heat for between 15 min and 20 min, stirring occasionally with a plastic covered rod.

Transfer the contents of the beaker into the same filter paper. Wash six times with the ammonium chloride solution (see 7.3.6), twice with hydrochloric acid (see 7.3.20) and twice with hot water. Add 10 ml of hydrochloric acid (see 7.3.18) to the first filtrate before carefully combining with the second filtrate and washings, stirring frequently to avoid the formation of a precipitate that is difficult to dissolve again.

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

Calculate the percentage of insoluble residue in the sample to the nearest 0.1 %, k , from the following formula:

$$k = \frac{R \times 100}{c}$$

where

R is the mass of dried residue;
 c is the sample mass.

NOTE 3 If the sample contains insoluble combustible material (e.g. carbon black), ignite the dried paper at (950 ± 5) °C in a pre-weighed crucible and re-weigh. Report the loss in mass as combustible material.

Evaporate the combined filtrates and washings to a volume of about 300 ml by boiling. 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 part hydrochloric acid + 1 part water), approximately 1 g of ashless filter powder and 5 ml polyethylene oxide solution (see 7.3.36), stirring thoroughly during and after each addition. Stand for 5 min and filter through a medium ashless filter paper. Quantitatively transfer all the solids to the filter using hydrochloric acid (see 7.3.20) and then wash thoroughly with hot water until free from acid.

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 at $(850 \pm 25)^\circ\text{C}$, and then at $(1\,200 \pm 25)^\circ\text{C}$ to constant mass. Weigh as soluble silica.

Calculate the percentage of soluble silica in the sample to the nearest 0.01 %, l , from the following formula:

$$l = \frac{m \times 100}{c}$$

where

m is the mass of ignited residue;

c is the sample mass.

Adjust the volume of the filtrate and washings to 500 ml (but see Note 5) in a graduated flask and use this prepared solution for other determinations as appropriate.

NOTE 4 This solution is referred to below as the stock test solution.

NOTE 5 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 sulfuric acid (1 part sulfuric acid + 1 part water). The residue is ignited and weighed as before. The residue is fused in the crucible with 0.5 g to 2 g potassium hydrogen sulfate. The cooled melt is dissolved in hot water containing a few drops of hydrogen chloride and added to the main filtrate before diluting to 500 ml. In this case the stock test solution cannot be used for the determination of sulfur trioxide.

NOTE 6 In the procedures for CaO , SO_3 , Fe_2O_3 , Al_2O_3 and MgO , the aliquots proposed are for a 5 g sample. If a 10 g sample is used, the necessary adjustment should be made to provide the equivalent aliquots.

7.5.3.4 Calcium oxide (CaO)

Pipette 5 ml of the stock test solution into a 500 ml conical flask and add 5 ml triethanolamine solution (see 7.3.46) and 10 ml of potassium hydroxide solution (see 7.3.38). 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 standard solution (see 7.3.16) from a semi-micro burette until the colour change is from fluorescent green to pink-purple.

Calculate the percentage of calcium oxide in the sample to the nearest 0.01 %, n , from the following formula:

$$n = \frac{T \times s \times 10}{c}$$

where

T is the titre EDTA used;

s is the CaO equivalent (mg);

c is the sample mass.

7.5.3.5 Sulfur trioxide (SO_3)

Pipette 100 ml of the stock test solution (but see Note 1 in 7.5.3.3) 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 (see 7.3.8) drop-wise 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 two to three drops of barium chloride solution, and leave to cool for 12 h to 24 h. Filter the precipitate on a fine filter paper 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.

NOTE 1 Some reduction of the barium sulfate might occur during ignition but by adding two to three drops of sulfuric acid (see 7.3.44) and re-igniting, re-oxidation occurs.

Calculate the percentage of sulfur trioxide in the sample to the nearest 0.01 %, o , from the following formula:

$$o = \frac{p \times 34.3 \times 5}{c}$$

where

- p is the precipitate mass (as $BaSO_4$);
 c is the sample mass.

NOTE 2 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 7.5.2.3 but with the procedure in Note 1 of that clause omitted.

7.5.3.6 Iron (as Fe_2O_3)

Transfer a 5 ml aliquot of the stock test solution to a 100 ml graduated flask. Add 2 ml of hydroxylammonium chloride solution (see 7.3.24) and 5 ml of freshly prepared 1,10-phenanthroline solution (see 7.3.33). Add successive 2 ml portions of ammonium acetate solution (see 7.3.5) 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 filter in a suitable instrument.

NOTE 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 (see 7.3.27).

Calculate the percentage of total iron in the sample to the nearest 0.10 %, q , from the following formula:

$$q = \frac{r \times 10}{c}$$

where

- r is the mg Fe_2O_3 in solution;
 c is the sample mass.

7.5.3.7 Aluminium oxide (Al_2O_3)

Pipette 50 ml of the stock test solution into a separating funnel and add 20 ml of sulfuric acid (1 part sulfuric acid + 1 part water) and cool. Add 20 ml of chloroform and 10 ml of filtered cupferron solution (see 7.3.13). Support the funnel and shake vigorously. Release the pressure in the funnel by carefully removing the stopper and rinse the stopper and neck of the funnel with water. Allow the layers to separate and withdraw the chloroform layer. Confirm that extraction is complete by checking that the addition of a few drops of cupferron solution to the aqueous solution does not produce a permanent coloured precipitate. If the aqueous solution does produce a permanent coloured precipitate, the procedure should be stopped and then repeated from the beginning.

Add further 10 ml portions of chloroform and repeat the extraction until the chloroform layer is colourless. Discard the chloroform extracts.

Run the aqueous solution from the cupferron-chloroform separation into a 1 000 ml conical flask. Add a few drops of bromophenol blue indicator and add ammonia solution (see 7.3.3) until just alkaline.

Re-acidify quickly with hydrochloric acid (see 7.3.18) and add five to six drops in excess.

Add sufficient DCTA standard solution (see 7.3.14) to provide an excess of a few millilitres over the expected amount. Then add ammonium acetate buffer solution until the indicator turns blue, followed by 10 ml in excess.

Add an equal volume of ethanol (see 7.3.17), 20 ml of hydroxylammonium chloride (see 7.3.24) and 1 ml to 2 ml of dithizone indicator, and titrate with standard zinc solution (see 7.3.48) from green to the first appearance of a permanent pink colour.

NOTE The end point is often improved by the addition of a little naphthol green B solution (7.3.31) to eliminate any pink colour that might be formed in the solution on the addition of the indicator.

If the DCTA standard solution is not exactly 0.05 M calculate the equivalent volume of 0.05 M DCTA.

Calculate the percentage of aluminium oxide in the sample to the nearest 0.01 %, t , from the following formula:

$$t = \frac{2.55 (V_6 - V_7)}{c}$$

where

V_6 is the volume of DCTA standard solution (0.05 M) (ml);

V_7 is the volume of standard zinc solution (0.05 M) used in the back-titration (ml);

c is the sample mass.

7.5.3.8 Magnesium oxide (MgO)

Pipette a 5 ml aliquot of the stock test solution into a 500 ml conical flask and dilute with water to about 100 ml. Add 20 ml of triethanolamine (1 part triethanolamine + 1 part water) and about 25 ml of ammonia solution (see 7.3.3). Dilute to about 200 ml with water and add about 0.15 g of methylthymol blue complexone indicator. Titrate with EDTA standard solution (see 7.3.16) from a semi-micro burette, until the colour changes from blue to colourless.

NOTE This titration is the sum of the calcium and magnesium titration.

Calculate the percentage of magnesium oxide in the sample to the nearest 0.01 %, u , from the following formula:

$$u = \frac{(V_3 - V_4) \times w \times 10}{c}$$

where

V_3 is the volume of EDTA standard solution used for the calcium and magnesium titration (ml);

V_4 is the volume of EDTA standard solution used in the calcium titration (ml);

w is the MgO equivalent (mg);

c is the sample mass.

7.5.3.9 Chloride (Cl^-)

Accurately weigh 10 g of the 600 μ m ground sample (see 7.4.5) into a stoppered conical flask. Dissolve with 50 ml water and add 10 ml nitric acid (see 7.3.32). Heat to near boiling and keep hot for between 10 min and 20 min. Cool and add a measured excess of standard 0.1 M silver nitrate. Add 2 ml trimethyl hexanol (nonyl alcohol), stopper the flask and shake vigorously to coagulate the precipitate. Add 1 ml iron III indicator solution and titrate with the approximately 0.1 M ammonium thiocyanate to the first permanent red colour. Standardize the ammonium thiocyanate against the silver nitrate by the same method as above.

Calculate the percentage of chloride in the sample to the nearest 0.01 %, y , from the following formula:

$$y = V_1 - \frac{V_2 K}{0.1} \times \frac{0.355}{c}$$

where

- V_1 is the volume of 0.1 M silver nitrate (AgNO_3) added;
- V_2 is the titre of ammonium thiocyanate ($\text{NH}_4 \text{SCN}$) solution;
- K is the molarity of ammonium thiocyanate ($\text{NH}_4 \text{SCN}$) solution;
- c is the sample mass.

8 Reports for chemical analysis

8.1 Report of analysis for ready-mixed lime: fine aggregate (sand) for mortar

The certificate for ready-mixed lime: fine aggregate for mortar shall include the following information:

- a) information from the certificate of sampling (see 5.3.4);
- b) method of preparing laboratory sample (see 7.4);
- c) free water, reported to the nearest 0.5 % (see 7.5.2.1);
- d) available lime reported to the nearest 0.1 %, as the minimum amount of lime that could have been present (7.5.2.2);
- e) if the analysis of the fine aggregate (sand) is known, the total lime content corrected for calcium compounds in the fine aggregate (sand), as $\text{Ca}(\text{OH})_2$, to the nearest 0.1 %;
- f) where available, the results of the analyses of constituent raw materials;
- g) description of deviations from sampling, sample preparation and test methods;
- h) comments on potential inaccuracy.

8.2 Report of analysis for cement-based mortars, plasters and renders

The certificate for cement-based mortars, plasters and renders shall include the following information:

- a) information from the certificate of sampling (see 5.3.4);
- b) method of preparing laboratory sample (see 7.4);
- c) fine aggregate (sand), silt and clay content, and aggregate grading when requested (see 7.5.3.2);
- d) insoluble residue, reported to the nearest 0.1 % (see 7.5.3.3);
- e) soluble silica (SiO_2), reported to the nearest 0.01 % (see 7.5.3.3);
- f) calcium oxide (CaO), reported to the nearest 0.01 % (see 7.5.3.4);
- g) where available, the results of the analyses of constituent raw materials;
- h) description of deviations from sampling, sample preparation and test methods;
- i) comments on potential inaccuracy.

NOTE Where mix proportions only are required, the analysis need not be reported beyond this point.

In some cases, the following tests are required to determine a possible cause of failure and these shall be reported as follows:

- 1) sulfur trioxide (SO_3), reported to the nearest 0.01 % (see 7.5.3.5);
- 2) total iron (as Fe_2O_3), reported to the nearest 0.01 % (see 7.5.3.6);
- 3) aluminium oxide (Al_2O_3), reported to the nearest 0.01 % (see 7.5.3.7);
- 4) magnesium oxide (MgO), reported to the nearest 0.01 % (see 7.5.3.8);
- 5) chloride (Cl^-), reported to the nearest 0.01 % (see 7.5.3.9);
- 6) bulk density of hardened material reported to the nearest 50 kg/m^3 .

8.3 Report of analysis for gypsum-based plasters

The certificate for gypsum-based plasters shall include the following information:

- a) information from the certificate of sampling (see 5.3.4);
- b) method of preparing laboratory sample (see 7.4);
- c) fine aggregate (sand), silt and clay content, and aggregate grading when requested (see 7.5.3.2);
- d) insoluble residue, reported to the nearest 0.1 % (see 7.5.3.3);
- e) soluble silica (SiO_2), reported to the nearest 0.01 % (see 7.5.3.3);
- f) calcium oxide (CaO), reported to the nearest 0.01 % (see 7.5.3.4);
- g) sulfur trioxide (SO_3), reported to the nearest 0.01 % (see 7.5.3.5);
- h) chloride (Cl^-), reported to the nearest 0.01 % (see 7.5.3.9);
- i) where available, the results of the analyses of constituent raw materials;
- j) description of deviations from sampling, sample preparation and test methods;
- k) comments on potential inaccuracy.

NOTE Where mix proportions only are required, the analysis need not be reported beyond this point.

In some cases, the following tests are required to determine a possible cause of failure and these shall be reported as follows:

- 1) magnesium oxide (MgO), reported to the nearest 0.01 % (see 7.5.3.8);
- 2) bulk density of hardened plaster, reported to the nearest 50 kg/m^3 .

9 Calculation of mix proportions and interpretation of results

9.1 General

Results of the chemical analysis shall be converted to the composition of the analysed sample and then to the original mix proportions. This shall be reported as a percentage by mass of the dry materials, i.e. unhydrated cement or gypsum, etc.

NOTE 1 The results obtained by chemical analysis on a mortar are normally for the hydrated form and possibly carbonated condition. To obtain the original mix proportions on the dry mass basis, the chemical analysis requires correcting for hydration and carbonation.

NOTE 2 A wide range of cement compositions are now in common use.

Wherever possible, the actual composition and bulk densities of the components from the supplier and use in the calculation of mix proportions shall be obtained.

NOTE 3 Only where the composition and bulk densities of the constituents are known can reliable volume proportions be calculated. Use of assumed data might give incorrect results which could, at times, be at variance with the true mix proportions.

If assumed data are used, the use of assumed data, and its potential for inaccuracy, shall be stated in the report.

NOTE 4 The precision to which the analyst is justified in reporting mortar compositions is much less than the precision of the analytical methods used. The analytical results can be used to indicate causes of failure. It should be remembered, however, that no problem should be investigated without full consideration being given to all information obtainable concerning the materials used, etc. In general the more information available, the more definite the conclusion of an investigation can be.

NOTE 5 The overlap of some of the figures in 9.2 and 9.3.3 is due to the wide variation in bulk densities of the various materials.

9.2 Ready-mixed lime: fine aggregate (sand) for mortar

9.2.1 Determination of volume proportions

If the analysis of the fine aggregate (sand) is known, determine the range of nominal proportions from inspection of Table 2 using the corrected total lime contents as Ca(OH)_2 (see 7.5.2.4).

Where the bulk densities of the constituents and the Ca(OH)_2 content of the lime are known, calculate the actual proportions by volume.

If the chemical analysis of the fine aggregate (sand) is not known, then the available lime shall be used instead.

Table 2 — Available lime contents of nominal volume mixes

Ca(OH) ₂ content of dry mass %	Nominal proportions of lime to fine aggregate (sand) by volume
9.0 to 14.0	1:3
6.5 to 10.5	1:4
4.5 to 9.5	1:4.5
3.5 to 8.0	1:6
2.0 to 5.5	1:9
1.5 to 4.0	1:12

NOTE If carbonation has occurred then the determination of available lime content (see 7.5.2.2) gives only a minimum value. A value of available lime below the lower figure given for a particular class of ready-mixed lime: fine aggregate (sand) does not necessarily mean that the mix did not conform to that class.

9.2.2 Report of composition of ready-mixed lime: fine aggregate (sand) for mortar

The complete certificate shall include the following information:

- information from the certificate of analysis (see 8.1);
- the range of equivalent nominal volume proportions with which the lime content corresponds, i.e. 1:3 to 1:4.5;
- where the actual bulk densities of the aggregate and lime are available, the volume proportions calculated and reported as:
 - one volume of lime to $A \pm 0.15A$, where A is the volume of aggregate;
 - report to the nearest 0.5 parts, giving the values of bulk density used.

NOTE Thus, for example, determined value 1:2.8, reported value $1:3 \pm 0.5$. Or determined 1:7.2, reported value $1:7 \pm 1$.

9.3 Freshly mixed mortars and plasters

NOTE In the case of freshly mixed materials, samples of the raw materials are made available. The cement is analysed for soluble silica (SiO₂) and calcium oxide (CaO). The aggregate is analysed for insoluble residue and soluble silica (SiO₂) and calcium oxide (CaO). (See 7.5.3.1, 7.5.3.3 and 7.5.3.4.)

9.3.1 Calculation of mix proportions

The aggregate content, other than for calcareous aggregates³⁾, shall be calculated as follows.

If the aggregate contains k % insoluble residue, then the percentage aggregate in mortar, z , shall be calculated using the following equation:

$$z = \frac{100A}{k}$$

where

A is the insoluble residue (obtained by analysis).

The soluble SiO₂ and CaO found to be present in the aggregate as required by 7.5.3.1 shall be subtracted proportionally from the total soluble SiO₂ and CaO obtained from chemical analysis.

If the aggregate contains B % soluble SiO₂, then the percentage soluble SiO₂ (or CaO) to be subtracted, C , is given by the equation:

$$C = \frac{z \times B}{100}$$

where

z is the percentage aggregate in mortar.

³⁾ Calcareous aggregates have an insoluble residue of less than 50 %. For these aggregates standardized calculation procedures are not given and the analyst should calculate the mix proportions using his/her own discretion.

If the aggregate contains F % soluble CaO, then the percentage soluble CaO to be subtracted, G , is given by the equation:

$$G = \frac{z \times F}{100}$$

where

z is the percentage aggregate in mortar.

The equations in Table 3, but with the figures determined for soluble SiO₂ for Portland cement and masonry cement, shall be used to calculate the composition. Where these equations are used, the potential for inaccuracy shall be included in the report.

Table 3 — Equations for the calculation of composition of freshly mixed or hardened mortars, plasters and renders

Cement: fine aggregate (sand)	Masonry cement: fine aggregate (sand)	Cement lime: fine aggregate (sand)	Gypsum: fine aggregate (sand)	Gypsum: lime: fine aggregate (sand)
$a = \frac{100B}{20.2}$ <p>where a is the percentage cement content, and B is the percentage soluble SiO₂ content</p>	$c = \frac{100B}{15.5}$ <p>where c is the percentage cement content, and B is the percentage soluble SiO₂ content</p>	$d = \frac{100B}{20.2}$ <p>where d is the percentage cement content, and B is the percentage soluble SiO₂ content</p>	$g = \frac{100L}{55.2}$ <p>where g is the percentage gypsum content, and L is the percentage SO₃ content</p>	$i = \frac{100L}{55.2}$ <p>where i is the percentage gypsum content, and L is the percentage SO₃ content</p>
$b = \frac{100F}{64.5}$ <p>where b is the percentage cement content, and F is the percentage of CaO</p>	See Note 3	$e = \frac{64.5d}{100}$ <p>where e is the percentage lime content</p>	$h = \frac{100F}{38.6}$ <p>where h is the percentage cement content, and F is the percentage lime content</p>	$j = \frac{38.6i}{100}$ <p>where j is the percentage lime content</p>
<p>a and b shall be within 1 % of each other If $a - b < 1$, the cement content shall be taken as $\frac{a+b}{2}$ If $a > b + 1$, soluble SiO₂ in the fine aggregate (sand) is indicated. If the fine aggregate (sand) is known to be free from CaCO₃ the cement content shall be taken as b. If $b > a + 1$, CaCO₃ in the fine aggregate (sand) is indicated; the cement content shall be taken as a.</p>	—	$f = \frac{100(F-e)}{72.7}$ <p>where f is the hydrated lime content</p>	<p>g and h should be within 1 % of each other. If $h > g + 1$, CaCO₃ in the fine aggregate (sand) is indicated; the gypsum content shall be taken as g.</p>	$k = \frac{100(F-j)}{727}$ <p>where k is the hydrated lime content</p>
<p>NOTE 1 The variables $a, b, c, d, e, f, g, h, i$ and j are not as defined in Clause 4. NOTE 2 These equations use assumed data about the composition of cement. Many different cement compositions are now permitted (see BS EN 197-1). Wherever possible the composition and bulk density of the cement should be obtained from the supplier. NOTE 3 The inclusion of lime in a masonry cement mortar can be established only when the analysis of the fine aggregate (sand) and the masonry cement is known. (See 9.4.4.) NOTE 4 The analyst should contact the product supplier for analytical data on raw materials.</p>				

Table 4 — Composition of types of mortars, plasters and renders: Cement: fine aggregate (sand) mortars and plasters (including air entrained)

Percentage by mass on dry mass mortar	Traditional volume proportioning	Traditional mortar designation
Portland cement %	Portland cement: fine aggregate (sand)	
20.5 to 25	1:3	<i>i</i>
16.0 to 25.0	1:3 to 4	<i>ii</i>
11.5 to 16.5	1:5 to 6	<i>iii</i>
8.5 to 12.5	1:7 to 8	<i>iv</i>
8.5 to 11.0	1:8	<i>v</i>

Table 5 — Composition of types of mortars, plasters and renders: Masonry cement: fine aggregate (sand) mortars and plasters

Percentage by mass on dry mass mortar	Traditional volume proportioning	Traditional mortar designation
Masonry cement %	Masonry cement: fine aggregate (sand)	
17.0 to 27.5	1:2.5 to 3.5	<i>ii</i>
12.5 to 19.0	1:4 to 5	<i>iii</i>
10.0 to 15.5	1:5.5 to 6.5	<i>iv</i>
9.5 to 12.5	1:6.5 to 7	n/a

Table 6 — Composition of types of mortars, plasters and renders: Cement: lime: fine aggregate (sand) mortars and plasters

Percentage by mass on dry mass mortar		Traditional volume proportioning	Traditional mortar designation
Portland cement %	Lime (Ca(OH) ₂) %	Portland cement: fine aggregate (sand)	
20.0 to 25.0	0 to 3.0	1:0 to 0.25:3	<i>i</i>
14.0 to 19.5	1.5 to 4.5	1:0.5:4 to 4.5	<i>ii</i>
11.0 to 15.5	3.0 to 7.0	1:1.5 to 6	<i>iii</i>
7.5 to 10.5	4.0 to 8.5	1:2:8 to 9	<i>iv</i>
5.5 to 8.0	6.0 to 10.0	1:3:10 to 12	<i>v</i>

Table 7 — Composition of types of mortars, plasters and renders: Gypsum: lime: fine aggregate (sand) plaster

Percentage by mass on dry mass plaster		Traditional volume proportioning
Gypsum (CaSO ₄ ·½ H ₂ O) %	Lime (Ca(OH) ₂) %	Gypsum: lime: fine aggregate (sand)
16.0 to 20.0	—	1:0:1.5
12.0 to 14.5	7.5 to 9.5	3:2:6
8.5 to 10.5	8.0 to 10.0	1:1:3
3.0 to 4.0	9.0 to 11.0	1:3:9

9.3.2 Interpretation of results

The values for fine aggregate (sand), silt, clay, cement (or gypsum) and lime (which normally total between 90 % and 96 %) shall be added. Each shall be corrected, by ratio, to total 100 %.

The type of binder (cement or gypsum) and whether lime is present shall be determined by inspection of the corrected results. Using the corrected values for the binder and lime (if present) refer to Table 4, Table 5, Table 6 and Table 7 for the designation or volume proportion.

NOTE 1 The figures in Table 4, Table 5, Table 6 and Table 7 have been calculated as follows. Aggregates are known to have dry bulk densities within the range 1 450 kg/m³ to 1 900 kg/m³. Although the dry bulk densities of cement, gypsum and hydrated lime vary, for the calculations here it is assumed that they are constant. See Table 8.

Table 8 — Dry bulk densities of material constituents

Constituent	Bulk density kg/m ³
Aggregate (dry)	1 450 to 1 900 ^a
Portland cement (BS EN 197-1:2000 CEM I Class 32.5, 42.5 and 52.5)	1 450
Air- entrained Portland cement (BS EN 197-1:2000 CEM I Class 32.5)	1 450
Portland limestone cement (BS EN 197-1:2000 CEM II A/L or A/LL Class 32.5, 42.5 and 52.5)	1 400
Portland fly ash cement (BS EN 197-1:2000 CEM II B-V Class 32.5 and 42.5)	1 400
Portland slag cement (BS EN 197-1:2000 CEM II B-S Class 32.5 and 42.5)	1 600
Sulfate resisting Portland cement (BS 4027:1996 Class 42.5)	1 450
Masonry cement (containing calcium carbonate) (BS EN 413-1:2004 Class MC 12.5)	1 375
Masonry cement (containing hydrated lime) (BS EN 413-1:2004 Class MC 12.5)	1 050
Hydrated lime	575
Gypsum plaster	860
^a In the absence of accurate data, assume 1 675 kg/m ³ .	

NOTE 2 Tables 4 and 5 use assumptions based on the properties of UK materials established up until 1994. The actual bulk density of materials from different suppliers varies from the values given and in addition, a much wider range of cements and limes have become available with the publication of CEN Standards for cements and limes. Cements and limes now in use in the UK may display different bulk densities from those given in Table 5 that will affect the calculation of mix proportions. Wherever possible, values for bulk density should be obtained from the supplier. It is essential that the value used be provided in the report. Consideration is to be given to including a comment that quantifies the effect on the stated prescribed mix proportions of variation in binder chemical composition and density.

NOTE 3 It is assumed in Table 3, Table 4, Table 5, Table 6, Table 7 and Table 8 that masonry cements contain 75 % Portland cement, and gypsum plasters contain 70 % of CaSO₄·½ H₂O. Using these figures, the composition range of original mixes on the dry mass of material can be calculated. The Portland cement clinker content of masonry cements can be down to 25 % so that, wherever possible, the composition should be obtained from the supplier.

If assumed data are used, the use of it and the potential for inaccuracy shall be stated in the report.

9.3.3 *Certificate of composition for freshly mixed mortars or plasters*

The certificate shall include the following information:

- a) information from the certificate of sampling (see 5.3.4);
- b) information from the certificate of analysis (see 8.2 and 8.3);
- c) the calculated composition of the mortar to the nearest 0.1 % for each constituent;
- d) the mortar, plaster designation, or traditional volume, proportioning from 9.3.1;
- e) any assumptions made in determining the mix proportions and interpretation (including those made in this British Standard).

9.4 Hardened mortars and plasters

9.4.1 *General*

Where the analyses of the raw materials are available, the results shall be treated as in 9.3.2.

Where analyses of the raw materials are not available, or where a failure has occurred, the results of chemical analysis reported in 8.2 and 8.3 shall be studied to determine whether the binder is cement or gypsum, using the following criteria.

- a) If the soluble silica content is high, e.g. above 0.5 %, assume the sample contains Portland cement.
- b) If the soluble silica content is low, e.g. less than 0.2 %, and the sulfate content is high, e.g. above 2 %, assume that the binder is gypsum.
- c) If both soluble silica and sulfate contents are below these values and the CaO content is above 5 %, assume that the binder is lime.

NOTE 1 Before reporting on the mix proportions, reference should be made to Annex A, which gives information relevant to the interpretation of the chemical analysis.

NOTE 2 The information in Annex A is based on the properties of established UK materials. A much wider range of cements and limes have become available with the publication of CEN Standards for cements and limes. Other values, particularly for soluble silica and calcium oxide, might need to be considered. The manufacturer of the material, if identified, should be consulted for information about material compositions. See 9.3.3.

9.4.2 *Calculation of mix proportions*

When the analysis of the aggregate is known to contain more than 50 % insoluble residue, the aggregate content shall be calculated as in 9.3.2. The soluble SiO₂ and CaO in this amount of aggregate shall be subtracted from the total soluble SiO₂ and CaO obtained from chemical analysis.

Calculate the composition of the mortar or plaster using the values in Table 4, Table 5, Table 6 and Table 7, but with the values determined for the actual constituents if these are known. When they are not available use the standard values given in Table 6.

NOTE 1 The values in Table 6 are based on the properties of UK materials established up until 1994. The actual values of materials from different suppliers might vary from the values given and, in addition, a much wider range of cements and limes have become available with the publication of CEN Standards for cements and limes. Cements and limes now in use in the UK might display different values to those given in Table 9, which affects the calculation of mix proportions. Wherever possible, values should be obtained from the supplier. It is essential that the value used is provided in the report.

Table 9 — Standard values for hardened mortars and plasters

	Constituent	Standard Value %	Typical Range %
Portland cement EXAMPLE 1 BS EN 197-1:2000 CEM I Class 32.5, 42.5 and 52.5	Soluble silica	20.2	19.5 – 21.0
	Calcium oxide	64.5	62.0 – 66.0
Air-entrained Portland cement EXAMPLE 2 BS EN 197-1:2000 CEM I Class 32.5	Soluble silica	20.2	19.5 – 21.0
	Calcium oxide	64.5	62.0 – 66.0
Portland limestone cement EXAMPLE 3 BS EN 197-1:2000 CEM II A/L or A/LL Class 32.5, 42.5 and 52.5	Soluble silica	—	17.0 – 20.0
	Calcium oxide	—	62.5 – 66.5
Portland fly ash cement EXAMPLE 4 BS EN 197-1:2000 CEM II B-V Class 32.5 and 42.5	Soluble silica	—	26.0 – 29.0
	Calcium oxide	—	43.5 – 51.5
Portland slag cement EXAMPLE 5 BS EN 197-1:2000 CEM II B-S Class 32.5 and 42.5	Soluble silica	—	23.0 – 26.0
	Calcium oxide	—	54.0 – 58.0
Sulphate resisting Portland cement EXAMPLE 6 BS 4027:1996 Class 42.5	Soluble silica	—	19.5 – 22.0
	Calcium oxide	—	61.5 – 65.5
Masonry cement (containing calcium carbonate and hydrated lime) EXAMPLE 7 BS 5224:1995 Class MC 12.5	If the source is known, it is strongly advised that guidance be sought from the supplier as different materials and amounts of filler are used by different suppliers. If the source is not known, a soluble silica content of 15.5 % shall be used for masonry cements applied prior to 1994.		
Hydrated lime EXAMPLE 8 BS EN 459-1:2001 CL90 EXAMPLE 9 BS EN 459-1:2001 CL80 EXAMPLE 10 BS EN 459-1:2001 CL70	Calcium oxide	72.7	65.6 – 75.6 ^a
		64.3	59.3 – 68.5 ^a
		56.7	52.7 – 62.6 ^a
Gypsum plaster (½ H ₂ O)	Sulfur trioxide	55.2	
	Calcium oxide	38.6	
^a Based on range permitted in BS EN 459-1:2001.			

When the analysis of the aggregate is not known but it is reasonable to assume that it contains no significant amounts of acid- or alkali-soluble material, the aggregate content shall be assumed to be equal to the insoluble residue and no corrections made to the total soluble SiO_2 and CaO obtained from chemical analysis. Some loss in confidence in the calculated composition of the mortar or plaster may occur. This shall be made clear in the certificate of composition.

NOTE 2 In other cases where the analysis of the aggregate is not known, make the best possible assumptions of its analysis. In such cases, the assumptions made, their justification and the effect on the accuracy of the calculated composition should be reported.

9.4.3 Interpretation of results

The type of mortar or plaster shall be analysed and determined using Table 4, Table 5, Table 6 and Table 7.

NOTE 1 When some or all of the determined cement, lime or gypsum contents from a batch of samples do not fall within any of the types designated in Table 4, Table 5, Table 6 and Table 7, an approximation of the nominal volume mix proportions can be calculated using the bulk densities of the ingredients given in 9.3.1.

The statement of the results shall not imply a greater accuracy in the calculated volume proportions than is justified by the assumption upon which it has been based. The assumptions made shall be included in the report.

NOTE 2 Estimates of repeatability and reproducibility of hardened mortar analysis are given in Annex B.

9.4.4 Certificate of composition for hardened mortar and plaster

The certificate of composition shall include the following information:

- a) information from the certificate of sampling (see 5.3.5);
- b) information from the certificate of analysis (8.2 or 8.3);
- c) the calculated composition of the mortar or plaster to the nearest 0.5 % for each constituent;
- d) the mortar or plaster designation of traditional volume proportioning from Table 4, Table 5, Table 6 and Table 7, or if outside this range, as calculated in 9.4.5;
- e) any assumptions made in calculations and interpretation (including those made in this British Standard).

NOTE Additional information concerning the reason for testing can also be reported.

Annex A (informative)

Information relevant to the calculations of mix proportions of hardened mortars and plasters

A.1 Iron oxide

A low iron oxide content (less than 0.1 %) indicates the use of white Portland cement, whereas a high iron oxide content (greater than 1 %) indicates the presence of iron salts in the aggregate or iron oxide colouring pigment.

A.2 Aluminium oxide

A high aluminium oxide content (greater than 0.5 %) indicates the presence of high alumina cement⁴⁾.

A.3 Magnesium oxide

A high magnesium oxide content (greater than 0.3 %) indicates the presence of magnesium lime.

A.4 Sulfur trioxide

NOTE Ordinary Portland cement mortars do not normally have an SO₃ to cement ratio of more than 4:100.

Analyses of cement-based mortar showing a SO₃ to cement ratio higher than 4:100 indicates the possibility of the mortar having suffered sulfate attack.

A.5 Chloride

Most mortars contain traces of chloride. Those found to contain more than 0.2 % of chloride ion by mass are likely to have been made with marine or coastal aggregates or with chloride containing admixtures. When the chloride ion content exceeds 0.2 % by mass, it is recommended that the specification for the mortar be checked for requirements relating to the use of admixtures and for the chloride content of the aggregate.

A.6 Portland cement

The characteristic analysis for Portland cement shows a ratio of calcium oxide to soluble silica of about 3:1.

NOTE 1 Where the calcium oxide content of the mortar is higher than would be indicated by this ratio, the mortar might have contained lime from calcareous aggregate.

A.6.1 Sulfate-resisting Portland cement

NOTE 2 Generally, the ratio of aluminium oxide to iron oxide is 1:1 or less in sulfate-resisting Portland cement. Because the amount of acid soluble aluminium and iron oxides in fine aggregates (sand(s)) varies so widely, a sample of the fine aggregate (sand) used in the preparation of the mortar is essential if any conclusion is to be drawn from the ratio of aluminium oxide to iron oxide in mortar. Even when such a sample is available it is unlikely, in view of the variation that might take place in the fine aggregate (sand) analysis from batch to batch, to be possible to show whether or not the cement conformed to the requirements of BS 4027. BS 1881-124 includes a method for distinguishing between sulfate-resisting Portland cement and Portland cement.

A.7 White Portland cement

This material is characterized by a ratio of aluminium oxide to iron oxide of about 20:1.

A.8 High alumina cement

Mortars made from high alumina cement are characterized, unless calcium carbonate has been added as a workability aid or filler, by the ratio of calcium oxide to aluminium oxide of about 1:1.

NOTE 1 The extraction procedure specified in this standard is not suitable for high alumina cement and the determined aluminium oxide content might be too low.

NOTE 2 Normally, lime should not be used with high alumina cement, and therefore excess calcium might be assumed to be derived from calcium carbonate in the aggregate, or added as filler.

A.9 Masonry cement

NOTE 1 Until 1994, masonry cements were Portland cements with a maximum of 25 % finely ground material (that may be increased to 35 % if it has hydraulic properties) together with an air-entraining agent. A much broader range of compositions is now permitted, including formulations based on Portland cement and lime.

⁴⁾ It is anticipated that BS 915-2, *Specification for high alumina content — Part 2: Metric units*, is to be superseded by prBS EN 14647, *Calcium aluminat cement — Composition, specifications and conformity criteria*, on the latter's publication.

If calcium carbonate or lime has been used, it is not possible to distinguish between masonry cement mortar and carbonated lime based mortar. If silica has been used, this will be shown by the high clay and silt content, and a microscopic examination of the residue from a clay determination will identify the clay fraction.

NOTE 2 If ground granulated blast furnace slag (ggbs) has been used, its presence might be established by a sulfide determination. An indication of its presence might be given by the hydrogen sulfide, recognizable by its smell, evolved from the mortar when treated with hydrochloric acid. Another possible filler is fly ash, which is also called pulverized fuel ash (pfa).

A.10 Pulverized fuel ash

The presence of fly ash (or, pfa) is shown by the microscopic examination of the residue after treatment of the mortar with salicylic acid in methanol.

The mortar shall be broken down by suitable means, the fine aggregate shall be removed to ensure an adequate concentration of the cement matrix, and the material passing a 150 μm sieve shall be collected. Stir 1 g with 7 g of salicylic acid and transfer the residue to a microscopic slide. Immerse in water and examine at a magnification of $\times 25$. Fly ash (or, pfa) contains characteristic spherical particles.

NOTE If the mortar contains fly ash (or, pfa), it might not be possible to obtain accurate mix proportions because of the solubilisation of silica with time.

A.11 Granulated blast furnace slag

Ground granulated blast furnace slag (ggbs) has a calcium oxide to soluble silica ratio of about 1 to 15:1. It also contains about 1 % manganese oxide.

NOTE Analysis of the mortar for these species might establish the presence of slag. Blast furnace slag is almost completely soluble in dilute acids and its presence is normally indicated by the generation of hydrogen sulfide.

Annex B (informative)

Estimates of precision in the analysis of hardened mortars

B.1 Precision data

Estimates of repeatability and reproducibility of hardened mortar analysis by the methods described in this standard are given in Table B.1 and Table B.2. The data has been obtained for mortars made with Portland cement or masonry cement and with siliceous fine aggregates (sand)s of known composition. The data might not be applicable to mortars made with other constituents.

Details of the precision experiment, the definitions of the terms used, and other precision data are given in B.2.

NOTE 1 The calculation of cement content from a determination of CaO is not applicable to mortars containing hydrated lime or fine aggregates (sand) with a high calcium content.

NOTE 2 It is not possible to determine the hydrated lime content of a hardened mortar made with a fine aggregate (sand) containing an unknown or high amount of calcium.

Table B.1 — Precision estimates for cement content expressed as a percentage of the mass of dry ingredients (fine aggregate (sand) corrections applied)

Oxide used to calculate cement content	Repeatability r_1 %	Reproducibility R_1 %	Reproducibility R_2 %
Soluble SiO_2	2.2	3.2	3.3
CaO (see Note 1 of B.1)	0.8	1.8	1.9

Table B.2 — Precision estimates for hydrated lime content expressed as a percentage mass of dry ingredients (fine aggregate (sand) corrections applied)

Repeatability r_1 %	Reproducibility R_1 %	Reproducibility R_2 %
2.1	3.0	3.0

B.2 The precision experiment, definition of terms and other precision data

B.2.1 The precision data given in Table B.1 and Table B.2 were obtained from an experiment conducted in 1986 and 1987 involving 15 laboratories. The experiment was organized, and the data analysed, according to BS 5497-1. The following types of mortar were analysed in the experiment:

- a) 1:1.5:7.7 ordinary Portland cement (now known as CEM-1 in BS EN 197-1) content 9.9 %, lime content 5.9 %;
- b) 1:5.8 masonry cement:fine aggregate (sand) mortar (MCS); cement content 12.7 %.

Samples of 2 kg mass were prepared. Two samples of each mortar were selected at random and sent to each laboratory. All samples were treated in accordance with BS 4551:1980 in order to yield two independent test portions for analysis. Control samples of the fine aggregate (sand)s used to make the mortars were distributed and tested. The results were used to correct the mortar analysis for CaO and soluble SiO₂ contribution from the fine aggregate (sand)s.

B.2.2 The test for outliers and stragglers, described in BS ISO 5725-2, were applied to the test results. A number were found as shown in Table B.6. The outliers were excluded from the calculation of the precision coefficients but the stragglers were included. Comparison of the precision coefficients obtained for the percentage of cement by mass on the three mortars indicated that the differences were largely due to the presence or absence of stragglers. It is therefore appropriate to calculate average values of the precision coefficients for use in practice, as given in Table B.1.

B.2.3 Confidence limits of 95 % for the average result obtained when one laboratory undertakes duplicate analyses of a single sample were calculated using the formula:

$$H \pm \sqrt{\frac{R_2^2}{2} - \frac{r_1^2}{4}}$$

where

- r_1 is the repeatability limit (see **B.2.4**);
- R_2 is a reproducibility limit (see **B.2.4**);
- H is the average result.

Values obtained using this formula are also shown in Table B.3 and Table B.4. Values appropriate to other circumstances may be calculated using the standard deviations given in the tables.

B.2.4 From the chemical analysis of hardened mortar, the following definitions of the repeatability conditions and repeatability limit, r_1 , and the reproducibility conditions, R_1 , and reproducibility limits, R_2 , apply.

- a) Repeatability conditions.

When independent test portions of the same sample of hardened mortar are analysed by the same operator, using the same equipment and within a short interval of time, in accordance with BS 4551.

- b) Repeatability limit, r_1 .

The difference between two test results obtained under repeatability conditions should exceed r_1 on only one in 20 occasions.

- c) Reproducibility limits.

When independent test portions of hardened mortar are analysed by different operators, using different equipment, in accordance with BS 4551.

- d) Reproducibility limits.

The difference between two test results obtained under reproducibility conditions should exceed R_1 or R_2 on only one in 20 occasions. When the test results are obtained on test portions from the same sample, R_1 applies. When they are obtained on test portions from different samples of the same batch of mortar, R_2 applies.

The standard deviations given in Table B.3 and Table B.4 are defined by the equations:

$$r_1 = 2.8 \sqrt{V_{r1}}$$

$$R_1 = 2.8 \sqrt{V_{r1} + V_L}$$

$$R_2 = 2.8 \sqrt{V_{r1} \pm V_L + V_s}$$

where

V is the variance (see Table B.3).

B.2.5 It is possible to convert the results of chemical analysis to volume proportions when the bulk densities of the ingredients are known. Table B.4 and Table B.5 give the precision data for the volumes of fine aggregate (sand) and hydrated lime, respectively, expressed as a ratio of their volume to one volume of cement.

Table B.3 — Precision data: analysis for percentage cement by mass — Control samples of fine aggregate (sand)s used

Analysis Method	Mortar	Number of laboratories	Overall average	Precision limits			Standard deviations			95 % confidence limits
				<i>Repeatability</i>	<i>Reproducibility</i>	<i>Reproducibility</i>	<i>Repeatability</i>	<i>Laboratories</i>	<i>Samples</i>	
		<i>n</i>	<i>X</i> %	<i>R</i> ₁ %	<i>R</i> ₁ %	<i>R</i> ₂ %	$\sqrt{V_{r1}}$ %	$\sqrt{V_L}$ %	$\sqrt{V_S}$ %	%
Soluble SiO ₂	OPCLS	15	9.6	2.8	3.1	3.1	0.99	0.46	0.00	—
	MCS	15	12.5	1.2	2.7	2.8	0.43	0.84	0.31	—
	Average	—	—	2.2	3.2	3.3	0.78	0.79	0.10	±2.1
CaO	MCS	15	12.9	0.8	1.8	2.0	0.27	0.58	0.27	—
	Average	—	—	0.8	1.8	1.9	0.29	0.56	0.23	±1.3

Table B.4 — Precision data: analysis for percentage lime by mass — Control samples of fine aggregate (sand)s used

Analysis Method	Mortar	Number of laboratories	Overall average	Precision limits			Standard deviations			95 % confidence limits
				<i>Repeatability</i>	<i>Reproducibility</i>	<i>Reproducibility</i>	<i>Repeatability</i>	<i>Laboratories</i>	<i>Samples</i>	
		<i>n</i>	<i>X</i> %	<i>R</i> ₁ %	<i>R</i> ₁ %	<i>R</i> ₂ %	$\sqrt{V_{r1}}$ %	$\sqrt{V_L}$ %	$\sqrt{V_S}$ %	%
CaO	OPCLS	15	5.5	2.1	3.0	3.0	0.75	0.76	0.00	±1.8

Table B.5 — Precision data: fine aggregate (sand) volume proportions to one volume of cement

Analysis method	Mortar	Number of laboratories	Overall average	Precision limits			Standard deviations			95 % confidence limits
				<i>Repeatability</i>	<i>Reproducibility</i>	<i>Reproducibility</i>	<i>Repeatability</i>	<i>Laboratories</i>	<i>Samples</i>	
Insoluble residue with		<i>n</i>	<i>X</i> %	<i>R</i> ₁ %	<i>R</i> ₁ %	<i>R</i> ₂ %	$\sqrt{V_{r1}}$ %	$\sqrt{V_L}$ %	$\sqrt{V_S}$ %	%
Soluble SiO ₂	OPCLS	15	7.7	2.8	2.7	2.7	0.83	0.45	0.00	±1.5
	MCS	15	5.7	0.4	1.0	1.1	0.16	0.31	0.18	±0.8
	Average	—	—	1.1	1.5	1.5	0.40	0.31	0.06	±2.1
CaO	MCS	15	5.5	0.2	0.7	0.9	0.08	0.26	0.17	—
	Average	—	—	0.2	0.6	0.7	0.09	0.20	0.12	±0.5

Table B.6 — Precision data: lime volume proportions to one volume of cement

Analysis Method	Mortar	Number of laboratories	Overall average	Precision limits			Standard deviations			95 % confidence limits
				<i>Repeatability</i>	<i>Reproducibility</i>	<i>Reproducibility</i>	<i>Repeatability</i>	<i>Laboratories</i>	<i>Samples</i>	
		<i>n</i>	<i>X</i> %	<i>R</i> ₁ %	<i>R</i> ₁ %	<i>R</i> ₂ %	$\sqrt{V_{r1}}$ %	$\sqrt{V_L}$ %	$\sqrt{V_S}$ %	%
CaO	OPCLS	15	1.5	0.8	1.0	1.0	0.30	0.19	0.00	±0.6

Table B.7 — Numbers of outliers and stragglers found in the precision experiment

Property	Analysis method	Mortar	Laboratory averages		Between-test portion ranges	
			Outliers	Stragglers	Outliers	Stragglers
% cement	Soluble SiO ₂	OPCLS	0	0	0	0
		MCS	0	0	0	0
% cement	CaO	MCS	0	0	1	2
% lime	CaO	OPCLS	0	0	0	3

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