

# Mortar — Methods of test for mortar and screed — 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

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| C1       | 31 May 2013     | Corrigendum to remove mark up instructions in subclause 9.4.4 |
| C2       | 30 June 2014    | Corrigendum to update the front cover and amendments table    |

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## Foreword

This British Standard is published by BSI Standards Limited, under licence from The British Standards Institution.

This British Standard has been prepared by Subcommittee B/519/2. BS 4551:2005+A2:2013 supersedes BS 4551:2005+A1:2010, which is withdrawn.

Text introduced or altered by Amendments No. 1 and No. 2, respectively, are indicated in the text by tags **A1** **A1** and **A2** **A2**. Minor editorial changes are not tagged.

The revision specifies the chemical analysis of mortar **A2** and screed **A2** 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 **A2** and screed **A2** only, and should not be used or quoted as a specification for mortar **A2** or screed **A2**. 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 **A2** and screed **A2** 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 **A2** or screed **A2** 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, **A2** plastering, rendering or screeding **A2**.

For this purpose, it should be realized 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 cannot 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,  $\text{A}_2$  screeding,  $\text{A}_2$  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  $\text{A}_1$  calcium aluminate  $\text{A}_1$  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.

$\text{A}_1$  BS 410-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*. [ISO 3310-1]  $\text{A}_1$

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*.

$\text{A}_1$  BS 6100-9, *Building civil engineering — Vocabulary — Part 9: Work with concrete and plaster*.  $\text{A}_1$

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  $\text{A}_1$  BS 6100-6.9  $\text{A}_1$  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 ( $\text{CaO}$  and/or  $\text{MgO}$ ) and/or hydroxide ( $\text{Ca(OH)}_2$  and/or  $\text{Mg(OH)}_2$ ) 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.

### **A2** 3.9 screed

mix of one or more inorganic binders, aggregates, water and sometimes admixtures and/or additions, used either to obtain a defined level or to carry a final flooring or to provide a wearing surface **A2**

## 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 $\text{SiO}_2$                                      |
| <i>b</i>                 | loss in mass (g)                                                       |
| <i>C</i>                 | percentage soluble $\text{SiO}_2$ (or $\text{CaO}$ ) 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 $\text{Ca(OH)}_2$ by wet mass          |
| <i>e</i>                 | percentage of available lime as $\text{C(OH)}_2$ by dry mass           |
| <i>F</i>                 | percentage soluble $\text{CaO}$                                        |
| <i>f</i>                 | percentage of available lime by wet mass                               |
| <i>G</i>                 | percentage soluble $\text{CaO}$ to be subtracted                       |
| <i>g</i>                 | total lime content as the percentage of $\text{CA(OH)}_2$ 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 <b>9.3.1</b> )                    |
| <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 ( $\text{NH}_4 \text{SCN}$ ) solution |
| <i>k</i>                 | percentage of insoluble residue in the sample                          |
| <i>L</i>                 | percentage $\text{SO}_3$                                               |
| <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 sulfur trioxide in the sample                            |
| <i>p</i>                 | precipitate mass (as $\text{BaSO}_4$ )                                 |
| <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> <sub>1</sub>    | reproducibility conditions                                             |
| <i>R</i> <sub>2</sub>    | reproducibility limit (see <b>B.2.4</b> )                              |

|       |                                                                                    |
|-------|------------------------------------------------------------------------------------|
| $r$   | mg Fe <sub>2</sub> O <sub>3</sub> in solution                                      |
| $r_1$ | is the repeatability limit (see B.2.4)                                             |
| $s$   | CaO equivalent of EDTA (mg)                                                        |
| $T$   | 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 <sub>3</sub> )                                |
| $V_2$ | titre of ammonium thiocyanate (NH <sub>4</sub> 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  $\square A_1$  dry  $\square A_1$  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 m<sup>2</sup> 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 <sup>2</sup> | Maximum area represented by each composite main sample |
|----------------------------------|-----------------------------------------------------------|--------------------------------------------------------|
|                                  | g                                                         | m <sup>2</sup>                                         |
| Masonry mortar                   | 50                                                        | 50                                                     |
| Plastering or rendering mortar   | 50                                                        | 50                                                     |
| <b>A2</b> Floor screed <b>A2</b> | <b>A2</b> 100 <b>A2</b>                                   | <b>A2</b> 100 <b>A2</b>                                |

### 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 **A1** by chipping away the mortar or by use of a dry core drill.

NOTE The use of a masonry drill or wet core drill might contaminate the sample or degrade it and these should not be used. **A1**

#### 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.

#### **A2** 5.3.3.3 Screeding mortar

Each sub-sample shall be a coherent piece of full depth. Take the sub-samples from regularly spaced positions over the area to be examined. When a failure of adhesion to the sub-base has occurred, break out appropriate sub-samples, using a hammer and cold chisel or bolster. Where adhesion is good, cut out or core the sub-samples. Examine the surface of the sub-base for the presence of grout and/or foreign matter. **A2**

### 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  $(10 \pm 0.5)$  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  $(25 \pm 0.1)$  mm, and a mass of  $(9.8 \pm 0.25)$  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  $(150 \pm 15)$  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  $45^\circ$  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).

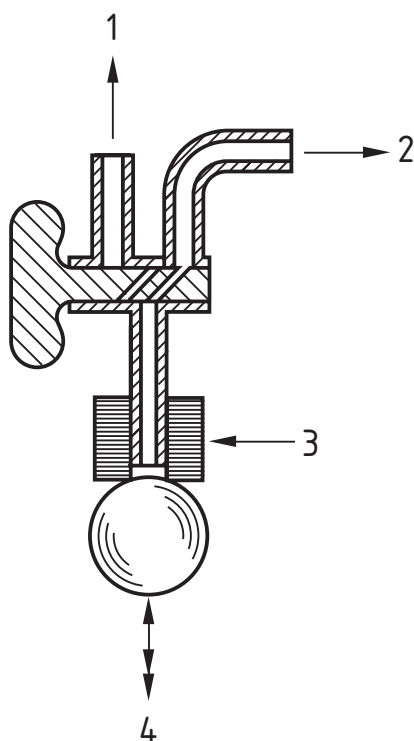
Three tests shall be conducted on separate portions of the mortar.

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+A1:2009;
- 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**

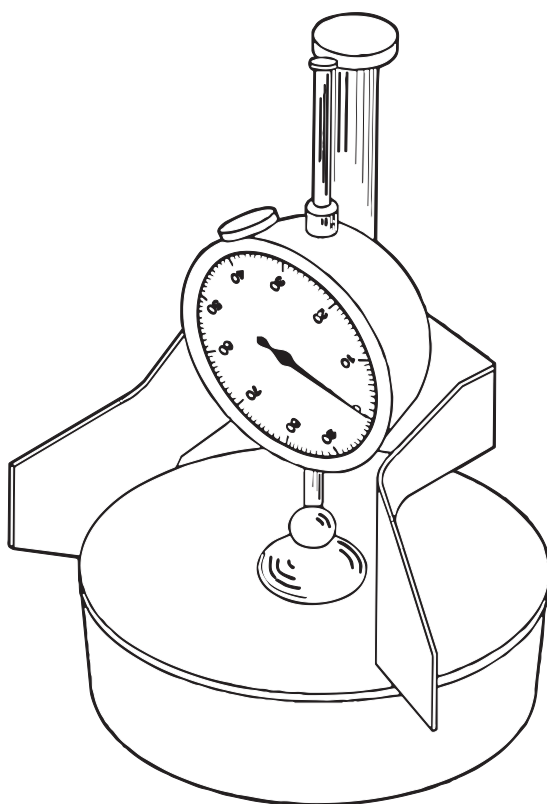


**Figure 2 — Suitable device for measuring penetration**

## 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/m<sup>2</sup>, 110 mm in diameter.*

### **6.2.3 Procedure**

Either test the mortar within  $(150 \pm 15)$  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.

**A1** 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.

Either:

- weigh the filter papers to the nearest 0.05 g and calculate the mass of water removed; or
- discard the filter papers and weigh the full mould, including cotton gauze, and calculate the mass of water removed (in which case it is unnecessary to first weigh the filter paper). **A1**

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+A1:2010;
- 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  $(850 \pm 25)^\circ\text{C}$ ,  $(925 \pm 25)^\circ\text{C}$  and  $(1\,200 \pm 25)^\circ\text{C}$ .

**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 ( $150^\circ\text{C}$ ) 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.