BS 1881-124:2015

BSI Standards Publication

Testing concrete –

Part 124: Methods for analysis of hardened concrete

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Foreword

Publishing information

This part of BS 1881 is published by BSI Standards Limited, under licence from The British Standards Institution, and came into effect on 31 August 2015. It was prepared by Subcommittee B/517/1, *Concrete production and testing*, under the authority of Technical Committee B/517, *Concrete and related products.* A list of organizations represented on this committee can be obtained on request to its secretary.

Supersession

This British Standard supersedes BS 1881-124:1988, which is withdrawn.

Information about this document

In 2012, in cooperation with eleven UKAS-accredited testing laboratories, the Concrete Society Working Group undertook two round-robin trials using the BS 1881-124:1988 [1] analysis methods for cement content, chloride content, sulfate content, alkali content and original water content based on the laboratories' accredited methods. The tested samples included hardened concrete made with CEM I only and concrete containing CEM I with GGBS or fly ash. The repeatability and reproducibility were significantly poorer than in a 1983 exercise and the Concrete Society Working Group recommended that the methods could continue to be used, but in full knowledge of the potential levels of inaccuracy. The findings of the 2012 round-robin trials are summarized in Concrete Society Technical Report 32 (second edition), *Analysis of hardened concrete: A guide to tests, procedures and interpretation of results* [*1*].

Relationship with other publications

The BS 1881 series contains test methods for concrete currently used in the United Kingdom which are not covered by BS EN 12350, BS EN 12390 and BS EN 12504. Reference is made to the relevant part of BS EN 12350, BS EN 12390 and BS EN 12504 where appropriate. These test methods may be used in conjunction with BS EN 206.

- BS 1881, *Testing concrete*, is published in the following parts:
- BS 1881-113, *Method for making and curing no-fines test cubes*;
- BS 1881-119, *Method for determination of compressive strength using portions of beams broken in flexure (equivalent cube method*);
- BS 1881-122, *Method for determination of water absorption*;
- BS 1881-124, *Methods for analysis of hardened concrete*;
- BS 1881-125, *Methods for mixing and sampling fresh concrete in the laboratory*;
- BS 1881-129, *Method for the determination of density of partially compacted semi-dry fresh concrete;*
- BS 1881-130*, Method for temperature-matched curing of concrete specimens;*
- BS 1881-131, *Methods for testing cement in a reference concrete*;
- BS 1881-204, *Recommendations on the use of electromagnetic covermeters*;
- BS 1881-206, *Recommendations for determination of strain in concrete*;
- BS 1881-207, *Recommendations for the assessment of concrete strength by near-to-surface tests*;

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[1] This has been superseded by this British Standard.

- BS 1881-208, *Recommendations for the determination of the initial surface absorption of concrete*;
- BS 1881-209, *Recommendations for the measurement of dynamic modulus of elasticity*;
- BS 1881-210, *Determination of the potential carbonation resistance of concrete – Accelerated carbonation method*

Hazard warnings

WARNING. Where skin is in contact with fresh concrete, skin irritations are likely to occur owing to the alkaline nature of cement. The abrasive effects of sand and aggregate in the concrete can aggravate the condition. Potential effects range from dry skin and irritant contact dermatitis, to severe burns in cases of prolonged exposure. Take precautions to avoid dry cement entering the eyes, mouth and nose when mixing mortar or concrete by wearing suitable protective clothing. Take care to prevent fresh concrete from entering boots and use working methods that do not require personnel to kneel in fresh concrete. Unlike heat burns, cement burns might not be felt until sometime after contact with fresh concrete, so there might be no warning of damage occurring. If cement or concrete enters the eye, immediately wash it out thoroughly with clean water and seek medical treatment without delay. Wash wet concrete off the skin immediately. Barrier creams may be used to supplement protective clothing but are not an alternative means of protection.

Use of this document

It has been assumed in the preparation of this British Standard that the execution of its provisions will be entrusted to appropriately qualified and experienced people, for whose use it has been produced.

Presentational conventions

The provisions of this standard are presented in roman (i.e. upright) type. Its methods are expressed as a set of instructions, a description, or in sentences in which the principal auxiliary verb is “shall”.

Commentary, explanation and general informative material is presented in smaller italic type, and does not constitute a normative element.

Contractual and legal considerations

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.

1 Scope

This part of BS 1881 describes sampling procedures, treatment of samples and analytical methods to determine the cement content, aggregate content, aggregate grading, original water content, type of cement, type of aggregate, chloride content, sulfate content and alkali content of a sample of concrete.

The procedures are applicable to concretes made with CEM I cements and, in favourable circumstances, concretes containing ground granulated blastfurnace slag (GGBS).

This part of BS 1881 does not cover the analysis of concretes made with other cements and the determination of fly ash content.

2 Normative references

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

BS 410-1 (ISO 3310-1), *Test sieves – Technical requirements and testing – Part 1: Test sieves of metal wire cloth*

BS 8500 (all parts), *Concrete – Complementary British Standard to BS EN 206*

BS EN 196-2:2013, *Methods of testing cement – Part 2: Chemical analysis of cement*

BS EN 196-7, *Methods of testing cement – Part 7: Methods of taking and preparing samples of cement*

BS EN 206, *Concrete – Specification, performance, production and conformity*

BS EN 450-1, *Fly ash for concrete – Part 1: Definition, specifications and conformity criteria*

BS EN 932-1, *Tests for general properties of aggregates – Part 1: Methods for sampling*

BS EN 934-2, *Admixtures for concrete, mortar and grout – Part 2: Concrete admixtures – Definitions, requirements, conformity, marking and labelling*

BS EN 12390-7, *Testing hardened concrete – Part 7: Density of hardened concrete*

BS EN 13263-1, *Silica fume for concrete – Part 1: Definitions, requirements and conformity criteria*

BS EN 15167-1, *Ground granulated blast furnace slag for use in concrete, mortar and grout – Part 1: Definitions, specifications and conformity criteria*

BS EN ISO 1042, *Laboratory glassware – One-mark volumetric flasks*

BS ISO 3310-2, *Test sieves – Technical requirements and testing – Part 2: Test sieves of perforated metal plate*

3 Terms and definitions

For the purposes of this part of BS 1881, the terms and definitions given in BS 8500 (all parts) and BS EN 206 apply.

4 Sampling

*COMMENTARY ON CLAUSE **4***
The tests in this part of BS 1881 are usually applied only when there is some uncertainty about the quality of the concrete (for example, the average quality of a mass of

concrete or the quality of a particular part of this mass might be in question). The size of the mass can vary greatly and the sampling techniques necessary to provide information on the quality of a concrete are determined by the form of the concrete (for example, the requirements for a concrete retaining wall and for a concrete backing to a decorative panel can be quite different). For these reasons it is extremely difficult to specify how a sample should be taken and no requirements can be given.

No sample should be taken for testing without agreement between the interested parties about the method of taking the sample and the quantity of material that is considered to be represented by the sample (see ***4.2****).*

4.1 Selection of sample

When selecting a concrete sample ensure that:

a) the minimum linear dimension of the sample is at least five times that of the nominal maximum aggregate size;

b) the sample is in a single piece and has no evidence of fracture cracking apparent, if original water content is to be determined;

c) the mass is not less than 1 kg in any case, not less than 2 kg if the original water content is to be determined, and not less than 4 kg if aggregate grading is to be determined;

NOTE The minimum mass of the sample is determined by testing requirements.

d) foreign matter and reinforcements are avoided unless they are subject to test;

e) the sample is indelibly labelled with full particulars, including the date, exact position from which the sample was taken, the method of sampling and any other relevant information; and

f) the sample is sealed or tied securely into a heavy-duty polyethylene bag.

4.2 Number of samples

When information about the composition of concrete with a volume of up to 6 m^3 is required, take not less than 2 (preferably 4) representative and independent samples and analyse them separately.

When very large volumes of concrete or large numbers of concrete units are to be examined, take not less than 10 independent samples and analyse them separately.

NOTE The results can then be used to identify locations requiring more extensive investigation.

4.3 Additional material

COMMENTARY ON ***4.3***
In addition to the concrete samples, information on the composition of all the constituent materials improves the accuracy of the test results.

Where they are available, take representative samples of all the constituents of the concrete used in accordance with the relevant British Standard (i.e. aggregates BS EN 932-1; cement BS EN 196-7; GGBS BS EN 15167-1; fly ash BS EN 450-1; silica fume BS EN 13263-1; admixture BS EN 934-2).

Enclose the samples in heavy-duty polyethylene bags that are clearly labelled and sealed or tied securely.

Where such samples are not available, provide any information on the source and nature of these materials that might be useful to the test(s) being carried out, such as acid-soluble material content in aggregates; soluble silica and calcium oxide contents in cement, aggregates and additions.

4.4 Documentation of sampling

With each sample provide a document signed by the person responsible for taking it, stating that sampling was carried out in accordance with this part of BS 1881 [2)] and including the following information:

a) the date and time of sampling;

b) a description of the structure or component under investigation;

c) the location of the positions from which the concrete samples were taken;

d) the method of sampling;

e) details of the concrete, in particular its age, where known;

f) the identification of the samples (including orientation, see **5.3**);

g) the name of the sampler; and

h) the signature of the person responsible for sampling.

5 Treatment of samples

5.1 Apparatus

5.1.1 *Ventilated oven,* controlled to maintain a temperature of (105 ±5) °C.

5.1.2 *Crushing and grinding equipment*.

NOTE Jaw crushers, hammer mills, disc grinders, gyratory mills and mechanical or hand-operated mortars and pestles are examples of suitable equipment.

5.1.3 *125 µm test sieve*, conforming to BS 410-1 (ISO 3310-1) or BS ISO 3310-2, complete with the appropriate size of lid and receiver.

*NOTE Similar test sieves with apertures of 4.0 mm, 2.0 mm and 500 µm are also required if mechanical crushing and grinding is not used (see **5.5.2**).*

5.1.4 *Permanent bar magnet.*

5.1.5 *Airtight bottles,* made of glass or plastics and with a capacity of 100 mL to 500 mL.

5.1.6 *Saw,* fitted with a diamond or carborundum blade, when original water content is to be measured.

5.1.7 *Device for sample reduction,* e.g. a riffle box.

5.2 Density

5.2.1 When the content of constituents, determined as a percentage by mass of the oven-dried (105 °C) concrete, is to be converted to kg/m^3 of concrete, measure the oven-dried (105 °C) density of a single sound piece of concrete before starting any work involving breakdown of the concrete samples.

5.2.2 Determine the volume (V_3) of the sample by water displacement and its oven-dried density (ρ_9) in accordance with BS EN 12390-7.

[2)] Marking BS 1881-124 on or in relation to a sample represents a declaration of conformity, i.e. a claim by or on behalf of the person responsible for sampling that the sampling meets the requirements of the standard. The accuracy of the claim is solely the claimant’s responsibility. Such a declaration is not to be confused with third-party certification of conformity.

5.3 Determination of original water content

5.3.1 Cut a slice approximately 20 mm thick to produce a single face area of not less than 10 000 mm^2 from the sample with two parallel cuts, preferably with a diamond saw (**5.1.6**), avoiding as far as possible loss of material out of the cut faces.

5.3.2 Saw from top to bottom of the concrete as cast where this can be identified.

NOTE For horizontally-drilled cores from columns, walls, etc., that are more than 1 m high, the slice may be cut parallel to the axis of the core to provide a specimen of adequate size.

5.3.3 Test samples that have a cross-sectional area of less than 2 500 mm^2, e.g. 50 mm cubes or 50 mm diameter cylinders or cores, for water content without sawing before the test for cement content.

5.3.4 To reduce carbonation of the concrete, store specimens in airtight containers until they are ready for testing.

5.4 Nature of aggregate

Examine the concrete sample (see **10.3**), and broadly classify both coarse and fine aggregate under the following headings:

a) type I: natural aggregates essentially insoluble in dilute hydrochloric acid;

b) type S: natural aggregates largely soluble in dilute hydrochloric acid; and

c) type O: other aggregates.

5.5 Preparation of test samples

5.5.1 Initial treatment

5.5.1.1 Break up the sample into lumps not larger than approximately 50 mm in size, avoiding as far as possible fracture of the aggregate.

5.5.1.2 Dry the lumps in the oven (**5.1.1**) at (105 ±5) °C for at least 15 h and continue until constant mass is achieved.

NOTE Constant mass is achieved when successive weighings after drying at least 1 h apart do not differ by more than 0.1%, or the mass does not change by more than 0.2% in any 24 h period.

5.5.1.3 Allow the lumps to cool to room temperature.

5.5.1.4 Divide the sample into two portions, A and B, so that portion A is approximately three-quarters of the bulk and portion B is the remainder, ensuring as far as possible that each portion is representative of the whole.

5.5.2 Treatment of portion A

*COMMENTARY ON **5.5.2***
Avoid the loss of material (particularly dust), during the crushing and grinding operations. Avoid excessive particle size reduction during the preparation of the analytical sample. The crushing, grinding and following operations should be carried out as quickly as possible so that the sample is exposed to atmospheric carbon dioxide for the minimum time.

A major source of error in the analysis of hardened concrete is inadequate sample preparation.

A suitable procedure to provide a representative sub-sample of not less than 20 g of ground material, all passing a 125 µm sieve, to be used as the analytical sample is as follows.

5.5.2.1 Using suitable crushing and grinding equipment (**5.1.2**), crush portion A until it all passes a 4.0 mm sieve (see **5.1.3**, Note).

5.5.2.2 Using a riffle box or by equivalent means (**5.1.7**), subdivide this to produce a sub-sample weighing between 500 g and 1 000 g.

5.5.2.3 Crush this sub-sample such that it can pass through a 2.0 mm sieve (see **5.1.3**, Note) and reduce by two separate subdividing operations, discarding one half on each occasion.

5.5.2.4 Crush the whole of the remaining part such that it can pass through a 500 μm sieve (see **5.1.3**, Note) and again reduce by two separate subdividing operations, discarding one half on each occasion.

5.5.2.5 Crush the whole of the remaining part such that it can pass through a 125 μm sieve (**5.1.3**).

NOTE 1 When mechanical means of crushing and grinding are used some of these stages may be bypassed. For example, the initial crushing of portion A might yield a product passing a 1 mm sieve. In this case the subdivision before the next stage should provide a sub-sample of between 100 g and 200 g. This may then all be ground in a suitable grinder to pass a 125 μm sieve.

NOTE 2 Before the final grinding operation, the material in the sub-sample which passes a 125 μm sieve should be removed by sieving, and reserved and recombined with the ground fraction. If a number of passes through the grinding apparatus is required, this removal of fine material should be done each time.

5.5.2.6 Remove any metallic iron from the analytical sample by placing it in a porcelain evaporating basin or similar container and then stirring with a permanent bar magnet (**5.1.4**).

5.5.2.7 Transfer the analytical sample to a clean dry bottle with an airtight closure (**5.1.5**) and thoroughly mix the sample by tumbling, rolling or shaking for at least 2 min.

5.5.2.8 When aggregate control samples are available, dry and grind them by the same method as that used for the concrete.

5.5.3 Treatment of portion B

Retain portion B in an airtight bottle for use in the tests in Clause **8**, Clause **10** and Clause **11**.

6 Cement and aggregate content

*COMMENTARY ON CLAUSE **6***
*The closeness of agreement between the cement and aggregate contents, determined by the methods for analysis described in this part of BS 1881, and the actual values depends on a knowledge of the chemical composition of these constituents of the concrete (see **6.4** to **6.8**).*

*If the procedure given in **6.5** is being used, the method for the extraction of soluble silica described in **6.5.1** should be followed exactly.*

Methods of proven accuracy are given for other determinations. Alternative methods for the determination of the following may be used:

*a) calcium oxide (see **6.5.4**), e.g. by atomic absorption spectrophotometry;*

*b) soluble silica (see **6.5.3**), e.g. by atomic absorption spectrophotometry;*

*c) loss on ignition (see **6.6**), e.g. by thermogravimetry;*

*d) carbon dioxide (see **6.7**), e.g. by instrumental methods; and*

*e) sulfide (see **6.8**), e.g. by instrumental methods,*

provided evidence is documented that such methods give equivalent results.

NOTE 1 The precision with which the analytical methods can be carried out, in terms of repeatability by one operator in one laboratory and reproducibility between different operators in different laboratories, has been determined for cement content (see Annex A). In the case of this precision experiment, the actual cement and aggregates used to make the concrete were available and the calculated mean cement contents were found to be close to the actual cement contents.

NOTE 2 In cases where samples of the original constituents of the concrete are not available for analysis, the agreement between observed and actual contents of cement and aggregate depends on the validity of the assumptions made on the composition of the materials present in the concrete sample (see ***6.9.8*** *and Annex B). For example, an error of 1% in the assumed calcium oxide content of an aggregate is equivalent to an error of about 35 kg/m^3 in the calculated cement content. Similarly, a 1% error in the assumed soluble silica content is equivalent to an error of approximately 110 kg/m^3.*

6.1 General

Use one or more of the methods detailed in this clause as appropriate.

Make duplicate analyses unless a series of concrete samples from a single batch is being tested. Carry out duplicate analyses from the sample preparation stage, i.e. two analytical samples (**5.5.1**) shall be prepared and each analysed separately.

Wherever possible, obtain representative samples of all the constituents used in making the concrete and carry chemical analyses on them in a similar manner to that described for the concrete analytical sample.

6.2 Reagents

6.2.1 General

Use reagents of analytical reagent quality and distilled or deionized water.

NOTE 1 Dilutions of acids and other liquid reagents are given as (1 + n) which indicates that one volume of the concentrated reagent is added to n volumes of water and mixed.

Filter solutions that are not transparent. Prepare the reagents in accordance with **6.2.2** to **6.2.18**.

NOTE 2 Proprietary reagents of equal quality may be used as alternatives.

6.2.2 Ammonium chloride solution, 1 g/L

Dissolve 1 g of ammonium chloride in 1 L of water.

6.2.3 Calcein (screened) indicator

Mix, by grinding together, 0.2 g of calcein, 0.12 g of thymolphthalein and 20 g of potassium chloride.

6.2.4 Calcium standard solution, approximately 1.00 g CaO/L

6.2.4.1 Weigh between 1.78 g and 1.79 g of pure calcium carbonate dried at a temperature in the range 150 °C to 200 °C and record the mass to the nearest 0.000 1 g.

6.2.4.2 Dissolve in a slight excess of dilute hydrochloric acid (1 + 4) (**6.2.9**), boil to expel carbon dioxide, cool and dilute to 1 L in a volumetric flask.

6.2.4.3 Express the concentration of CaO in g/L to three significant figures as mass of $CaCO_3$/1.784 8.

6.2.5 Triethanolamine solution (1 + 4)

6.2.6 Sodium hydroxide solution, 200 g/L

Dissolve 200 g of sodium hydroxide in 1 L of water.

6.2.7 EDTA standard solution

6.2.7.1 Dissolve 6.67 g of EDTA (ethylenediaminetetraacetic acid, disodium dihydrate salt) in warm water, filter if necessary, cool and dilute to 1 L. Store in a polyethylene bottle.

6.2.7.2 To standardize against standard calcium solution pipette 25 mL of the calcium standard solution (**6.2.4**) into a 250 mL beaker or flask, add 10 mL of triethanolamine solution (**6.2.5**) and 10 mL of sodium hydroxide solution (**6.2.6**), and dilute to about 150 mL.

6.2.7.3 Add approximately 0.15 g calcein indicator (**6.2.3**) and titrate with the EDTA solution until the fluorescent green of the indicator completely changes to pink-purple with no residual fluorescence.

NOTE The end-point is more easily seen if observed against a black background.

6.2.7.4 Calculate the CaO equivalent of the EDTA, E, (in mg CaO/mL) from the expression:

$$E = \frac{25 \times h}{t}$$

where:

h is the concentration of CaO in the standard calcium solution (in g/L);

t is the volume of EDTA required in the titration, to the nearest 0.05 mL.

6.2.8 Hydrochloric acid, relative density 1.18

6.2.9 Hydrochloric acid, dilute, (1 + 1), (1 + 4), (1 + 9) and (1 + 49)

6.2.10 Polyacrylamide solution, 1 g/L

Add 0.1 g of polyacrylamide, molecular weight about 5×10^6, to 100 mL of water. Stir using a mechanical stirrer until dissolved (see **6.3.1**, Note).

6.2.11 Polyethylene oxide solution, 2.5 g/L

Slowly add 0.5 g of polyethylene oxide, molecular weight less than 1×10^6, to 200 mL of water, stirring using a mechanical stirrer until dissolved. Discard after two weeks.

6.2.12 Sodium carbonate solution, 50 g/L

Dissolve 50 g of anhydrous sodium carbonate in 1 L of water.

6.2.13 Orthophosphoric acid, relative density 1.7

NOTE This is used when carbon dioxide is to be determined.

6.2.14 Starch solution

Suspend 1 g of starch in 5 mL of cold water and add to 100 mL of boiling water. After cooling, add a solution of 1 g of sodium carbonate in 10 mL of water and 3 g of potassium iodide.

NOTE This is used when sulfur as sulfide is to be determined.

6.2.15 Potassium iodate standard solution

NOTE This is used when sulfur as sulfide is to be determined.

6.2.15.1 Dry pure potassium iodate at (120 ±5) °C, and allow to cool in a desiccator.

6.2.15.2 Dissolve between 1.112 g and 1.113 g of the potassium iodate with 12 g of potassium iodide and two pellets of sodium hydroxide in freshly boiled and cooled water.

6.2.15.3 Dilute the solution to 1 000 mL in a volumetric flask, mix and store in a glass-stoppered bottle. 1 mL of this solution is equivalent to 0.5 mg sulfide.

NOTE This potassium iodate solution is stable for several months.

6.2.16 Sodium thiosulfate solution

NOTE This is used when sulfur as sulfide is to be determined.

6.2.16.1 Dissolve 7.4 g of sodium thiosulfate ($Na_2S_2O_3.5H_2O$) in water and make up to 1 L. Standardize this solution in accordance with **6.2.16.2** to **6.2.16.4**.

6.2.16.2 Pipette 20 mL of the potassium iodate standard solution (**6.2.15**) into a 500 mL beaker or flask, dilute to approximately 300 mL with water and add 40 mL of dilute hydrochloric acid (1 + 1) (**6.2.9**).

6.2.16.3 Immediately titrate with the sodium thiosulfate solution until the colour of the liquid becomes a pale yellow and then add 2 mL of the starch solution (**6.2.14**). Continue the titration until the solution changes from blue to colourless.

6.2.16.4 Record the volume, V, of sodium thiosulfate solution required to the nearest 0.01 mL.

6.2.16.5 Calculate the factor, T, of the sodium thiosulfate solution from the expression:

$$T = \frac{20}{V}$$

6.2.17 Stannous chloride, solid ($SnCl_2.2H_2O$)

NOTE This is used when sulfur as sulfide is to be determined.

6.2.18 Ammoniacal zinc sulfate solution

NOTE This is used when sulfur as sulfide is to be determined.

6.2.18.6 Dissolve 50 g of zinc sulfate ($ZnSO_4.7H_2O$) in 150 mL of water and 350 mL of concentrated ammonium hydroxide solution.

6.2.18.7 Allow to stand for at least 24 h and filter.

6.3 Apparatus

6.3.1 *General apparatus*, comprising the apparatus listed in **6.3.1.1** to **6.3.1.6**.

NOTE It is assumed that laboratories carrying out these tests are equipped with basic apparatus such as analytical balances, beakers, volumetric flasks, pipettes, burettes, filtration apparatus, magnetic and mechanical stirrers, etc. Only apparatus needed *specifically for the determinations necessary to analyse hardened concrete is therefore listed. The filter papers used should be ashless.*

6.3.1.1 *Volumetric glassware*, with an accuracy of class B or better in accordance with BS EN ISO 1042.

6.3.1.2 *250 mL polypropylene beakers*.

6.3.1.3 *Plastic stirring rods.*

6.3.1.4 *Furnace(s)*, controlled to maintain temperatures of (925 ±25) °C and (1 200 ±50) °C.

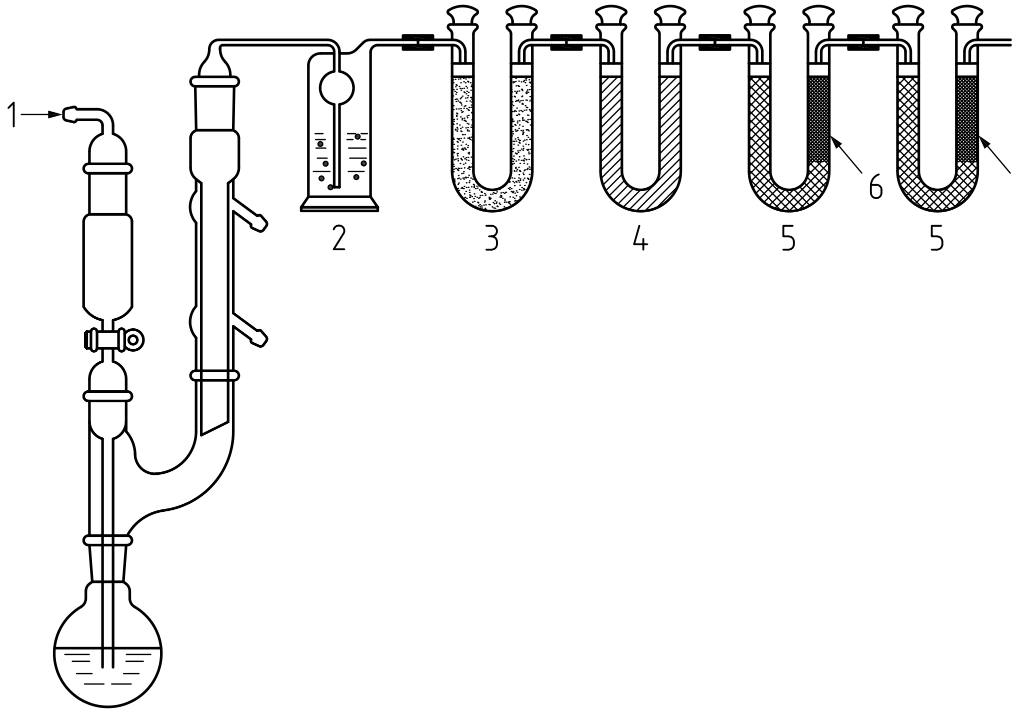
6.3.1.5 *Boiling water or steam bath*.

6.3.1.6 *Desiccator*, containing dried magnesium perchlorate.

6.3.2 *Apparatus for the determination of carbon dioxide,* which shall be free from leaks.

NOTE A typical apparatus is shown in Figure 1.

Figure 1 **Typical apparatus for determination of carbon dioxide content**



The apparatus comprises a reaction flask fitted with a tap funnel through which carbon dioxide free air or nitrogen can be passed at a controlled rate. The gas leaving the flask passes through a water cooled condenser, then through a bubbler containing concentrated sulfuric acid and then through a series of absorption tubes which successively contain pumice coated with anhydrous copper sulfate, dried magnesium perchlorate and then two weighable tubes both three quarters filled with a granular absorbent for carbon dioxide and one quarter filled with dried magnesium perchlorate.

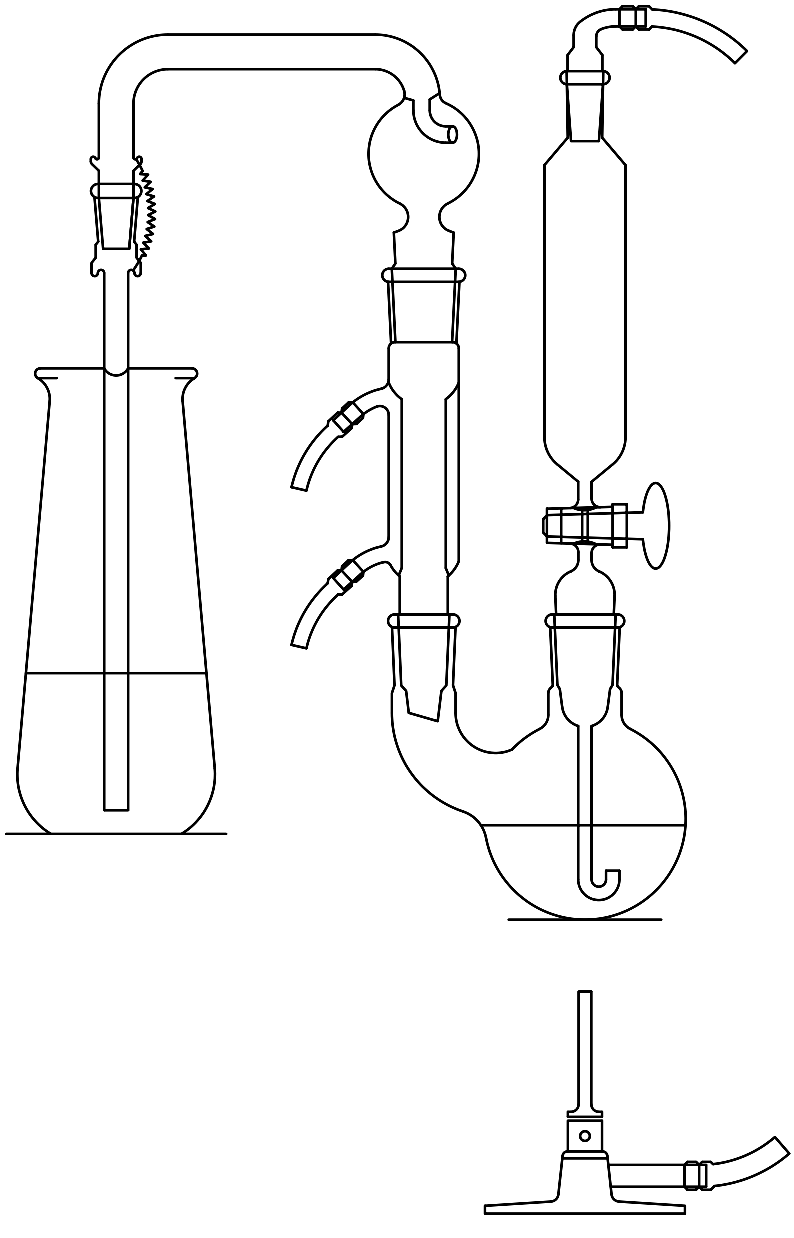
Key

1	CO_2-free gas	4	Dried $Mg(ClO_4)_2$
2	Conc. H_2SO_4	5	Granular absorbent for CO_2
3	Anhydrous $CuSO_4$	6	$MgO(ClO_4)_2$

6.3.3 *Apparatus for the determination of sulfide,* set up as follows. Fit the neck of a 250 mL round bottom flask with a ground-joint assembly to provide a means of adding acid and of passing air or other inert gas through the solution in the flask, an anti-splash device or a condenser and a delivery tube for the issuing gas. The flexible connecting tubes shall be made of sulfur-free material, polyvinyl chloride or silicone rubber.

NOTE A typical apparatus is shown in Figure 2.

Figure 2 **Typical apparatus for determination of content of sulfur as sulfide**



The delivery tube should dip into the solution in a 400 mL beaker. Regulation of the gas supply is facilitated by a bubbler consisting of a triple-necked Woolf flask, with its centre-neck fitted with a long glass tube dipping into water or into lead acetate solution (2 g/L), if the gas is likely to contain hydrogen sulfide or sulfur dioxide.

6.4 Determination of calcium oxide when insoluble residue and soluble silica do not need to be determined

6.4.1 Weigh (5 ±0.005) g of the analytical sample as prepared in **5.5.2** and transfer into a 500 mL beaker.

6.4.2 Disperse with between 50 mL and 100 mL of cold water, add 10 mL of concentrated hydrochloric acid (**6.2.8**), swirl, immediately add hot water to about 150 mL bring to the boil and keep hot for 5 min.

6.4.3 After cooling, transfer the solution quantitatively to a 500 mL volumetric flask, dilute to the mark with water and mix thoroughly.

6.4.4 Filter a portion of the solution through a dry medium ashless filter paper (see **6.3.1**, Note) or, alternatively, use a centrifuge to obtain a clear solution.

6.4.5 Pipette a 25 mL aliquot of the clear solution into a 250 mL beaker or flask and dilute to about 150 mL.

6.4.6 Add 10 mL of triethanolamine solution (**6.2.5**), 10 mL of sodium hydroxide solution (**6.2.6**) and about 0.15 g of screened calcein indicator (**6.2.3**).

6.4.7 Titrate with EDTA standard solution (**6.2.7**) using the magnetic stirrer until the indicator completely changes from fluorescent green to pink-purple with no residual fluorescence (see **6.2.7**).

6.4.8 Record the volume, V_1, of EDTA used in the titration to the nearest 0.05 mL.

*NOTE The EDTA solution and the use of potassium hydroxide, as described in BS EN 196-2:2013, **4.2.47**, are acceptable alternatives when determining calcium oxide. Other indicators which give clear end-points, e.g. HSN, may be used.*

6.4.9 Calculate the calcium oxide content, D, of the analytical sample, as a percentage, to the nearest 0.1%, from the expression:

$$D = \frac{2V_1E}{M_a}$$

where:

V_1 is the volume of EDTA solution used in the titration (in mL);

M_a is the mass of the analytical sample (in g);

E is the calcium oxide equivalent of the EDTA solution (in mg CaO/mL).

6.5 Determination of insoluble residue, soluble silica and calcium oxide

6.5.1 Extraction of soluble silica and preparation of stock test solution

6.5.1.1 Weigh (5 ±0.005) g of the analytical sample prepared in accordance with **5.5.2** into a 250 mL polypropylene beaker and carefully add 100 mL of dilute hydrochloric acid (1 + 9) (**6.2.9**).

6.5.1.2 When a limestone aggregate is known to be present or when vigorous effervescence on adding the acid indicates that it is likely to be present, add a further 10 mL of concentrated hydrochloric acid (**6.2.8**) to the stirred suspension.

6.5.1.3 Using a suitable mechanical stirrer such as a magnetic stirrer with a plastic covered magnetic follower, stir the suspension at room temperature for 20 min.

6.5.1.4 Allow to settle and decant the liquid through either a medium ashless filter paper (see **6.3.1**, Note) or a paper pulp pad supported on a perforated cone or similar, with or without suction.

6.5.1.5 Wash the residue in the beaker with three 25 mL portions of dilute hydrochloric acid (1 + 49) (**6.2.9**), pouring the washings through the same filter paper.

6.5.1.6 Retain this first filtrate.

*NOTE The rate of filtration is improved by adding 1 mL of polyacrylamide solution (**6.2.10**) to the acid extraction just before the completion of the 20 min stirring. The subsequent determinations based on the filtered extract are not affected. A coarser filter paper may be used.*

6.5.1.7 Place the filter paper plus any residue it contains back into the beaker containing the residue and add 100 mL of sodium carbonate solution (**6.2.12**).

6.5.1.8 Place the beaker on a boiling water or steam bath (**6.3.1.5**) for 15 min, stirring occasionally with a plastic rod to break up the filter paper.

6.5.1.9 Transfer the contents of the beaker on to a medium hardened ashless filter paper (see **6.3.1**, Note) or a paper pulp pad supported on a perforated cone or similar, and recover all the residue adhering to the sides of the beaker and the stirrer blade or magnet.

6.5.1.10 Wash six times with hot ammonium chloride solution (**6.2.2**), twice with hot dilute hydrochloric acid (1 + 49) (**6.2.9**) and twice with hot water, the volume of each of the washings being about 25 mL.

6.5.1.11 Allow each washing to drain before continuing with the next. Retain this second filtrate and the washings.

NOTE To reduce the possibility of silica being introduced into the solution, the filtrate should be collected in a plastic beaker.

6.5.1.12 Add 10 mL of concentrated hydrochloric acid (**6.2.8**) 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.

6.5.1.13 Reserve the combined filtrates for treatment as described in **6.5.3**.

6.5.2 Determination of insoluble residue

6.5.2.1 Place the filter paper containing the residue in a weighed crucible and ignite, at first slowly until the carbon of the paper is completely consumed without flaming and finally at (925 ±25) °C until constant mass is achieved (see **6.6**).

6.5.2.2 Allow the crucible and residue to cool to room temperature in the desiccator (**6.3.1.6**), weigh them and record the mass.

6.5.2.3 Calculate the insoluble residue content of the analytical sample as a percentage with an accuracy of 0.1% from the expression:

$$\text{insoluble residue} = \frac{\text{mass of ignited residue}}{\text{mass of analytical sample}} \times 100$$

6.5.3 Determination of soluble silica

6.5.3.1 Evaporate the combined filtrates and washings on a boiling water or steam bath (**6.3.1.5**) until not more than approximately 5 mL remain.

6.5.3.2 Add 10 mL of dilute hydrochloric acid (1 + 1) (**6.2.9**), approximately 0.5 g of ashless powder and 5 mL of the polyethylene oxide solution (**6.2.11**), stirring thoroughly during and after each addition.

6.5.3.3 Stand for 5 min and filter through a medium ashless filter paper (see **6.3.1**, Note).

6.5.3.4 Quantitatively transfer all the solids to the filter and wash thoroughly with hot water.

6.5.3.5 Adjust the volume of the filtrate and washings to 500 mL in a volumetric flask, and mix thoroughly to give the stock test solution for treatment as described in **6.5.4**.

NOTE The stock test solution may also be used for other determinations as required, e.g. sulfate, magnesia.

6.5.3.6 Place the filter paper containing the precipitate in a weighed platinum crucible and ignite, at first slowly until the carbon of the paper is completely consumed without flaming and finally at (1 200 ±50) °C until constant mass is achieved (see **6.6**).

6.5.3.7 Allow the crucible and residue to cool to room temperature in the desiccator and weigh them.

6.5.3.8 Calculate the soluble silica content of the analytical sample as a percentage to the nearest 0.1% from the expression:

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

*NOTE The purity of the ignited silica precipitate can be checked by evaporating it in the crucible with 10 mL of hydrofluoric acid containing 5 drops of sulfuric acid (1 + 1) in a fume cupboard. Ignite the residue and weigh as before. The loss in mass after evaporation and igniting equals the mass of silica in the original ignited residue. When the two masses differ by more than 0.005 g the determination (**6.5.3**) should be repeated. If the discrepancy persists, use the mass of silica found by evaporation with hydrofluoric acid to calculate the soluble silica content of the analytical sample. When determining soluble silica content, the concentration of hydrofluoric acid should be 40%, and it should be 60% when etching for microscopy.*

6.5.4 Determination of calcium oxide

6.5.4.1 Pipette 25 mL of the stock test solution (see **6.5.3.5**) into a 250 mL beaker or flask and dilute to approximately 150 mL. Add 10 mL of dilute triethanolamine solution (**6.2.5**), 10 mL of sodium hydroxide solution (**6.2.6**) and approximately 0.15 g of screened calcein indicator (**6.2.3**).

6.5.4.2 Titrate with the EDTA standard solution (**6.2.7**) and calculate the calcium oxide content of the analytical sample in accordance with **6.4**.

6.6 Determination of loss on ignition

6.6.1 Weigh between 1 g and 2 g of the analytical sample prepared in accordance with **5.5.2** into a crucible which has been previously ignited and weighed.

6.6.2 Place the crucible in a furnace (**6.3.1.4**) and, to avoid mechanical loss of material, cover the crucible with a lid, and raise the temperature slowly to (925 ±25) °C.

6.6.3 After 5 min at this temperature, remove the lid and leave the crucible in the furnace at (925 ±25) °C for a further 30 min.

6.6.4 Allow the crucible to cool to room temperature in the desiccator (**6.3.1.6**) and weigh.

6.6.5 Repeat the ignition, cooling and weighing (**6.6.2** to **6.6.4**) until constant mass is achieved as shown by successive weighings giving a difference in mass of less than 0.000 5 g.

6.6.6 Calculate the loss on ignition of the analytical sample as a percentage to the nearest 0.1% from the expression:

$$\text{loss on ignition} = \frac{(\text{mass of analytical sample} - \text{mass of ignited sample})}{\text{mass of analytical sample}} \times 100$$

6.7 Determination of carbon dioxide content

6.7.1 To determine the blank of the apparatus, which should be less than 1 mg, carry out the procedure in **6.7.2** to **6.7.9** with no sample in place. If the blank is greater than 1 mg, check the joints and ensure that they are free of leaks. Then repeat the blank determination. If a value greater than 1 mg is still obtained, replace the absorbents and repeat.

6.7.2 Assemble the apparatus (**6.3.2**) without the weighable tubes and flush it with gas free from carbon dioxide at a rate of approximately 3 bubbles per second for 15 min.

6.7.3 Ensure that the tap funnel is acid-free. Weigh approximately 0.2 g of the analytical sample prepared in accordance with **5.5.2** into a duplicate reaction flask, replace the empty reaction flask with the one containing the sample and continue to pass gas free from carbon dioxide.

6.7.4 Weigh the two absorption tubes and connect to the apparatus.

6.7.5 Disconnect the gas inlet and place 30 mL of concentrated orthophosphoric acid (**6.2.13**) into the tap funnel.

6.7.6 Open the tap and reconnect the gas inlet so that the gas pressure forces the acid into the reaction flask.

6.7.7 After effervescence has ceased, slowly raise the temperature of the contents of the flask to boiling and boil for 5 min.

6.7.8 Allow to cool for 15 min while maintaining the gas flow.

6.7.9 Detach the absorption tubes, transfer to the balance case and weigh after 30 min.

6.7.10 Calculate the carbon dioxide content of the analytical sample as a percentage to the nearest 0.1% from the expression:

$$CO_2 = \frac{\text{gain in mass of absorption tubes} - \text{blank}}{\text{mass of analytical sample}} \times 100$$

6.8 Determination of sulfide

*COMMENTARY ON **6.8***
This determination is only made when the slag content of a concrete containing a ground granulated blastfurnace slag (GGBS) of known composition as a reactive addition is required. When other constituents of the concrete contain sulfides, the method should not be used. Occasionally, when analysing hardened concrete, not all the sulfide is recovered and this can be substantial. The test therefore only determines the minimum ggbs content. When this occurs, it is shown by a significant difference between the CEM I contents subsequently determined from the calcium oxide content and from the soluble silica content, for concretes not containing significant quantities of calcium oxide in the aggregate, or determined from the aluminium oxide content and from the soluble silica content, for concretes containing significant quantities of calcium oxide in the aggregate.

6.8.1 Assemble the apparatus described in **6.3.3**.

6.8.2 Place 15 mL of ammoniacal zinc sulfate solution (**6.2.18**) and 285 mL of water in the beaker.

6.8.3 Weigh out 5.0 g of the analytical sample as prepared in **5.5.2** into the round bottom flask, add 2.5 g of stannous chloride (**6.2.17**) and disperse in 50 mL of water.

6.8.4 Ensure that the glass outlet tube is immersed in the zinc sulfate solution.

6.8.5 Using the separating funnel add about 50 mL of dilute hydrochloric acid (1 + 1) (**6.2.9**), and carefully close the tap of the separating funnel.

6.8.6 Connect the inert gas supply, open the tap and adjust the flow to about 10 mL/min.

6.8.7 Heat the contents of the flask to boiling and boil gently for between 5 min and 6 min.

6.8.8 Stop heating and leave for between 5 min and 6 min before shutting off the gas supply.

6.8.9 Disconnect the delivery tube, which then serves as a stirrer.

6.8.10 After cooling the contents of the beaker to approximately 20 °C, pipette into the beaker an excess of volume of standard potassium iodate solution (**6.2.15**), 20 mL is sufficient when the sample contains up to about 0.15% sulfide, and record the volume added, V_2, to the nearest 0.1 mL.

6.8.11 Add 40 mL dilute hydrochloric acid (1 + 1) (**6.2.9**), mix and immediately titrate with sodium thiosulfate solution (**6.2.16**) until the colour of the liquid becomes a pale yellow. Then add 2 mL of starch solution (**6.2.14**) and continue the titration until the colour changes from blue to colourless.

6.8.12 Record the volume of sodium thiosulfate solution required, V_4, to the nearest 0.1 mL.

6.8.13 Calculate the sulfide content, S, of the analytical sample as a percentage to the nearest 0.01 % from the expression:

$$S = \frac{(V_2 - V_4 T)}{20 M_\mathrm{b}}$$

where:

V_2 is the volume of standard potassium iodate solution (in mL);

V_4 is the volume of sodium thiosulfate solution used in the titration (in mL);

T is the factor from the standardization of the sodium thiosulfate solution;

M_b is the mass of the analytical sample (in g).

6.9 Calculation of cement and aggregate content

6.9.1 Determine the calcium oxide content (see **6.4** or **6.5.4**) and the soluble silica content (see **6.5.3**) of the analytical sample as prepared in **5.5.2** and, where available, of the ground aggregate sample and of the cement, and calculate the cement contents using first the calcium oxide determinations and then the soluble silica determinations.

6.9.2 When the two cement contents obtained from **6.9.1** are within 1% *(m/m)* of each other or, when converted to kg/m^3, are within 25 kg/m^3, report the mean value. When the two cement contents differ by more than this amount investigate the reasons for the discrepancy.

6.9.3 If no reason can be found, report both results and indicate which is to be preferred.

NOTE 1 This preference should be based on the analyst's experience, the age and exposure of the concrete and the nature of the aggregate, etc. Analysis of the sample for other constituents, such as insoluble residue, carbon dioxide, magnesium oxide, etc., *might prove helpful in making the assessment. If such an assessment is not possible, indicate that the lower cement content is to be preferred.*

NOTE 2 Where it is known that the aggregate has a calcium oxide content of less than 0.5%, the cement content should be calculated by way of the soluble silica content, and the cement content may be based on the result of the determination of the calcium oxide content alone. Conversely, where the calcium oxide content of the aggregate is 35% or more, calculations based on calcium oxide content should not be used.

6.9.4 Where the calcium oxide content of the aggregate is 35% or more and the soluble silica content of the aggregate is 10% or more, analyse the analytical sample, as prepared in **5.5.2**, for another constituent known to be present in substantially different amounts in the cement and in the aggregate and calculate the cement content by an appropriate modification of the equations in **6.9.6**.

6.9.5 If possible, the constituent chosen shall generally form a greater proportion of the cement than of the aggregate.

6.9.6 Using determined values for cement and aggregate and assuming that the combined water of hydration of the concrete is $0.23 \times C_1$ and the total mass of oven dried concrete consists of C_1% cement + F% aggregate + $0.23C_1$% combined water of hydration, calculate the cement content or the aggregate/cement ratio from the following expressions:

a) the percentage cement content, C_1, to the nearest 0.1%:

$$C_1 = \frac{c-b}{a-1.23b} \times 100$$

b) the cement content, C_2, to the nearest 1 kg/m^3:

$$C_2 = \frac{C_1 \times \text{oven-dried density of concrete}}{100}$$

c) the percentage aggregate content, F, to the nearest 0.1%:

$$F = \frac{a-1.23c}{a-1.23b} \times 100$$

d) the aggregate/cement ratio, A, to the nearest 0.1:

$$A = \frac{F}{C}$$

where:

a is the calcium oxide or soluble silica content of the cement (in %);

b is the calcium oxide or soluble silica content of the aggregate (in %) (see Note);

c is the calcium oxide or soluble silica content of the analytical sample (in %).

*NOTE If the overall aggregate grading is not to be determined (see Clause **8**), the best practical estimate of the relative proportions of the various sizes or supplies of aggregate in the concrete should be made. This may be done, for example, by visual inspection of the concrete or by consideration of the mix design. The weighted mean calcium oxide or soluble silica content of the aggregate should be calculated and these values used in the equations.*

6.9.7 If a ground granulated blastfurnace slag of known composition, e.g. *d*% calcium oxide, *e*% soluble silica and *f*% sulfide, has been incorporated in the concrete as a reactive addition, but the concrete does not contain blastfurnace slag aggregate and the sulfide content of the analytical sample has been determined (see **6.8**), then assuming no sulfide in the CEM I cement, calculate the percentage slag content, P, from the expression:

$$P = \frac{S}{f} \times 100$$

where:

S is the sulfide content.

The total calcium oxide content of the analytical sample is then corrected by subtracting:

$$\frac{P \times d}{100}$$

and the soluble silica content of the analytical sample is then corrected by subtracting:

$$\frac{P \times e}{100}$$

to give the calcium oxide or soluble silica content derived from the CEM I cement and aggregate, and these corrected values are used to calculate the CEM I cement content.

NOTE Where pulverized-fuel ash or natural pozzolana has been incorporated in the concrete as a reactive addition it is not possible to determine the content of the addition. However, where this material has a low calcium oxide content, e.g less than 2%, the analysis will permit the approximate estimation of the CEM I cement content provided the aggregate has an equally low calcium oxide content.

6.9.8 Using assumed values for cement and/or aggregate, determine the calcium oxide (see **6.5.4**) and soluble silica (see **6.5.3**) contents of the analytical sample as prepared in **5.5.2**. Make the best assumptions for the analysis of the cement and/or aggregate and calculate the cement content using the expressions in **6.9.6**.

NOTE Annex B indicates typical values for calcium oxide and soluble silica for types of cement currently available in the UK. For CEM I cements conforming to BS EN 197-1 there is little loss in accuracy in assuming that the calcium oxide content is 64.5% but rather greater loss in accuracy in assuming that the silica content is 20.2%. For some aggregates it might be possible to assume that the silica content is zero and for those essentially insoluble in hydrochloric acid it may be possible to assume that the calcium oxide content is zero.

6.9.9 Report the assumed values for the cement and aggregate analyses used in the calculations and, if possible, their justification.

7 Alternative method for aggregate content based on insoluble residue values

COMMENTARY ON CLAUSE ***7***
This test cannot be used if a cement, e.g. CEM II/B-V cement, having a significant insoluble residue (more than 10%) has been used.

Determine the insoluble residue (see **6.5.2**) of the analytical sample as prepared in **5.5.2**, and of the aggregate sample(s). Then, assuming no insoluble residue in the cement, calculate the aggregate content as a percentage from the expression:

$$\text{aggregate content} = \frac{\text{insoluble residue of analytical sample}}{\text{insoluble residue of aggregate}} \times 100$$

*NOTE When samples of the aggregate are not available for analysis there might be little loss of accuracy in assuming that with essentially insoluble aggregate the percentage insoluble residue of the analytical sample (see **6.5.2**) corresponds to the percentage aggregate content.*

8 Aggregate grading

*COMMENTARY ON CLAUSE **8***
This aggregate grading test is only applicable to concrete containing aggregates essentially insoluble in dilute hydrochloric acid.

8.1 Reagents

*NOTE See **6.2.1**.*

8.1.1 *Ammonium chloride solution,* 1 g/L (see **6.2.2**).

8.1.2 *Hydrochloric acid*, dilute (1 + 9) (see **6.2.9**).

8.1.3 *Polyethylene oxide solution,* 2.5 g/L (see **6.2.11**).

8.1.4 *Sodium carbonate solution*, 100 g/L. 100 g of anhydrous sodium carbonate dissolved in 1 L of water.

8.2 Apparatus

8.2.1 *4.0 mm test sieve*, conforming to BS 410-1 (ISO 3310-1) fitted with a lid and receiver.

8.2.2 *63 μm test sieve*, conforming to BS 410-1 (ISO 3310-1).

NOTE The sieves specified in BS EN 933-1 for sieving aggregate finer than 4.0 mm might also be required.

8.2.3 *Chisel-edged hand tool.*

NOTE Small electrically operated hand tools might be of assistance.

8.2.4 *Device for sample reduction,* such as a riffle box.

8.2.5 *1 L glass beakers*.

8.2.6 *Furnace,* controlled to maintain a temperature of (400 ±50) °C (see **8.3.2**, Note).

8.2.7 *Container,* of nickel, stainless steel or refractory material of a size large enough to hold the sample and to fit in the furnace (see **8.3.2**, Note).

8.3 Procedure

8.3.1 Use the sub-sample portion B obtained in accordance with **5.5.3**.

8.3.2 Separate the weighed sub-sample of concrete into coarse and fine fractions using the 4.0 mm sieve (**8.2.1**).

*NOTE Break-down of the concrete without fracture of the aggregate may be assisted by heating in a furnace (**8.2.6**) to (400 ±50) °C for 1 h to 16 h in a nickel, stainless steel or refractory container (**8.2.7**). If the matrix is still insufficiently weakened, thoroughly soak the concrete in water and re-heat. Microwave heating might also be appropriate.*

8.3.3 Carefully clean the coarse aggregate portion of adhering matrix by chipping the individual pieces with the chisel-edged tool (**8.2.3**). Pass the material removed through the 4.0 mm sieve.

8.3.4 When all the material larger than 4.0 mm has been cleaned, treat it with dilute hydrochloric acid (1 + 9) (**8.1.2**) until the aggregate is free from adhering cement, rinse with water, dry, weigh and record the mass, m_1.

8.3.5 Rinse with water the fine aggregate released during the acid cleaning of the coarse aggregate, dry and combine with all the material passed through the 4.0 mm sieve during the mechanical cleaning of the coarse aggregate.

8.3.6 Weigh and record the mass, m_2.

8.3.7 Using a riffle box or equivalent (**8.2.4**), subdivide the weighed combined material passing the 4.0 mm sieve to produce a sub-sample of between 50 g and 100 g.

8.3.8 Weigh and record the mass, m_3.

8.3.9 Transfer the weighed sub-sample, m_3, to a 1 L beaker (**8.2.5**), add 500 mL of dilute hydrochloric acid (1 + 9) (**8.1.2**) and heat to approximately 50 °C.

8.3.10 Maintain at this temperature, with occasional stirring, until disintegration appears to be complete. Add, whilst stirring, 5 mL of the polyethylene oxide solution (**8.1.3**).

8.3.11 Allow to settle and decant the liquid through a pre-weighed fast hardened ashless filter paper (see **6.3.1**, Note).

8.3.12 Wash the aggregate remaining in the beaker by decantation three times with water, each washing having a volume of 100 mL to 200 mL and being allowed to drain completely before the next.

8.3.13 Wash any residue on the paper back into the original beaker, dilute to 100 mL with water and add 100 mL of the sodium carbonate solution (**8.1.4**).

8.3.14 Simmer gently for 15 min avoiding any loss by frothing.

8.3.15 Transfer the contents through the 63 µm test sieve (**8.2.2**) into the original filter paper. Wash six times with ammonium chloride solution (**8.1.1**), twice with dilute hydrochloric acid (1 + 9) (**8.1.2**) and twice with hot water.

8.3.16 When the coarse aggregate is visibly different from the fine aggregate, examine the cleaned fine aggregate and extract those particles of fractured coarse aggregate which have obviously been introduced during the mechanical cleaning steps (**8.3.3**).

8.3.17 Dry the sieve residue and the filter paper plus its contents separately, at (105 ±5) °C, allow them to cool and weigh them separately.

8.3.18 Subtract the mass of the filter paper to give the mass of its contents and add this to the mass of the sieve residue to give the mass of recovered fine aggregate, m_4.

NOTE If required, a sieve analysis using the sieves specified in BS EN 933-1 may be carried out on the dried fine aggregate.

8.4 Calculations

8.4.1 Fine aggregate grading

Calculate the cumulative percentage passing each of the sieves used, including that with a 63 µm aperture, expressing the mass passing each sieve as a percentage of m_4.

NOTE Owing to the nature of the separation and sieving procedures used, the grading, particularly with respect to the fraction passing the 63 µm sieve, cannot be used to assess conformity of the grading of the fine aggregate with BS EN 12620.

8.4.2 Coarse aggregate grading

Do not use separated coarse aggregate to assess conformity of the grading of the coarse aggregate.

8.4.3 Overall aggregate proportions

Calculate the mass of fine aggregate in the sub-sample of concrete portion B as $(m_4 \times m_2)/m_3$. From this mass and the mass of separated cleaned coarse aggregate, m_1, calculate the relative proportions of coarse and fine aggregate.

9 Original water content

9.1 Applicability

*COMMENTARY ON **9.1***
The test described in this clause only gives acceptable results if the concrete is sound and not physically or chemically damaged. It is not suitable for poorly-compacted or semi-dry compacted concrete, air-entrained concrete or aerated concrete. For some aggregates, often manufactured, which are exceptionally porous or contain appreciable amounts of combined water, the corrections that would be applied are so great they would render the results of doubtful value.

The application of the method to concretes containing hydraulic binders other than CEM I cement is outside the scope of this British Standard.

Unreliable results might be obtained when concretes older than five years are tested owing to changes in the pore structure of such concretes.

The term "original water content" refers to the water present in the concrete mix at the time of setting.

The procedure involves the determination of:

a) the capillary porosity of the concrete originally filled with water at the time of setting; and

b) the combined water of hydration present in the prepared concrete sample.

The sum of these two represents the original water content of the concrete. It is usually expressed as the water/cement ratio and this entails the determination of the cement content of the prepared sample.

The prepared sample, while representative for the water/cement ratio of the whole sample, might not be representative for the cement content.

The water/cement ratio may be expressed in two ways:

1) as total water/cement ratio, which includes the water absorbed by the aggregates at the time of setting; or

2) as free water/cement ratio, which excludes the water absorbed by the aggregates at the time of setting.

The values for capillary porosity and combined water content include contributions from the aggregates present and these should be taken into account.

*When aggregate control samples are available, the correction for the combined water content of the aggregate should be made (see **9.5.5**).*

*Correcting for aggregate porosity (see **9.5.4**) gives the original free water/cement ratio, while not making the correction gives the original total water/cement ratio, provided that the aggregate pores were filled with water when the concrete had set. When there is some uncertainty about the condition of saturation of the aggregate in the set concrete, make the correction for aggregate porosity and report only the original free water/cement ratio. When there is no uncertainty, report both free and total water/cement ratios.*

When aggregate control samples are not available, assume that the combined water of hydration of the concrete is:

$0.23 \times C_1$

The range of values found for the combined water of hydration of BS EN 197-1 CEM I and CEM III cements is between 0.20 and 0.25 of the cement, for full hydration. For very young concretes (less than 28 days) special considerations might apply.

Take the capillary porosity of the aggregate to be equivalent to the water absorption value of the aggregate when this is reliably known (see **9.6**) to give the original free water/cement ratio. Otherwise make no correction for aggregate porosity and report only the total water/cement ratio on the assumption that the aggregate pores were filled with water when the concrete had set.

9.2 Reagent

9.2.1 *Tetrachlorethylene*.

NOTE 1,1,1- trichlorethane referenced in the 1988 edition of this British Standard is no longer available as an industrial chemical in the UK or wider EU. Tetrachlorethylene is the currently suggested alternative. However no published precision data are available for the use of this solvent in this test.

9.3 Apparatus

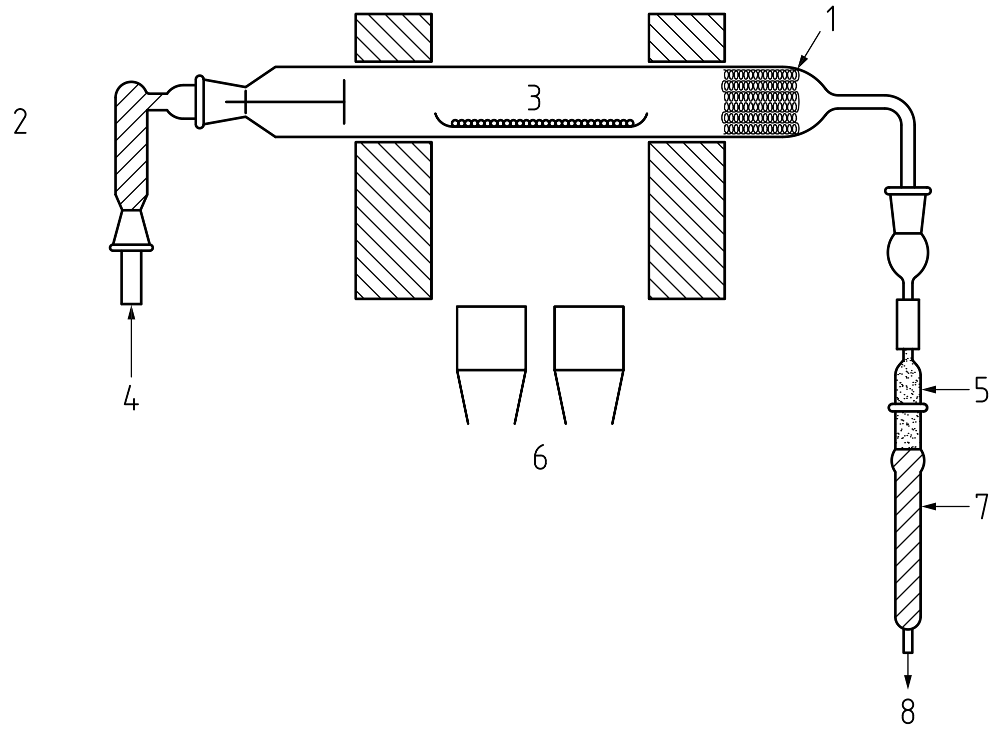
9.3.1 *Vacuum desiccator,* 250 mm diameter.

9.3.2 *Water pump or vacuum pump,* capable of reducing the pressure in the vacuum desiccator to less than 13.5 kPa (100 mmHg).

9.3.3 *Apparatus for the determination of combined water content,* consisting of a small absorption vessel, containing dried magnesium perchlorate, through which air, nitrogen or other suitable gas is passed before entering a silica combustion tube that is heated in a furnace capable of reaching 1 000 °C, bright red heat. The exit from the combustion tube shall be packed with silver wool to remove any hydrogen sulfide evolved. If silicone rubber connections are used it is advisable to protect them with heat reflectors. The gas leaving the combustion tube is passed through two weighable absorption tubes. The first quarter of each absorption tube is packed with silica gel and the remainder with dried magnesium perchlorate. A bubbler containing sulfuric acid is fitted after the exit end of the second absorption tube to allow the gas flow to be observed.

NOTE A typical apparatus is shown in Figure 3.

Figure 3 **Typical apparatus for determination of water combined content**



Key

1	Silver wool	5	Indicating silica gel
2	Dried $Mg(ClO_4)_2$	6	Burners
3	Sample	7	Dried $Mg(ClO_4)_2$
4	Pre-dried gas	8	To second absorber and bubbler

9.4 Determination of capillary porosity

WARNING. Avoid breathing the vapour and contact of the liquid with the skin and eyes. Wear gloves when handling the samples. Polyethylene gloves are recommended.

9.4.1 Prepare the sample by cutting a slice as described in **5.3**, drying it at (105 ±5) °C for not less than 16 h and allowing it to cool to room temperature in the desiccator (**6.3.1.6**).

9.4.2 Weigh the dried slice and immerse in tetrachlorethylene (**9.2.1**) contained in the vacuum desiccator (**9.3.1**).

NOTE 1,1,1-trichlorethane may be used if stocks are available.

9.4.3 Reduce the pressure in the vessel to less than 13.5 kPa (100 mmHg) by means of the water pump or vacuum pump (**9.3.2**).

NOTE It might be necessary to protect the vacuum pump against the effects of solvent vapour by fitting a cold trap or similar device.

9.4.4 Remove the air from the concrete.

NOTE 1 The air evolves from the capillaries in the concrete, rapidly at first and then more slowly. The removal of the capillary air is complete if no further air is released even when the evacuated vessel is cautiously given a sharp tap.

NOTE 2 The time required to remove the air might be several hours. "Boiling" of the tetrachlorethylene or 1,1,1-trichlorethane induced by low pressure with continued evacuation should not be confused with air evolution and is normally recognizable by the tendency of large bubbles to appear at a few points on the concrete surface.

9.4.5 When the air evolution has ceased, release the vacuum and keep the sample immersed at atmospheric pressure for a further 5 min.

9.4.6 Remove the sample from the reagent. Allow excess superficial liquid to drain away and quickly wipe excess solvent from the surfaces of the sample.

9.4.7 Immediately place the saturated sample in a weighed polyethylene bag, seal to prevent loss of liquid by evaporation, and weigh.

9.4.8 Calculate the mass of the reagent required to fill the pores of the concrete and from this mass derive the equivalent mass of water assuming that the relative density of tetrachlorethylene is 1.62 and 1,1,1-trichlorethane is 1.33.

9.4.9 Calculate the capillary porosity, *Q*, as a percentage to the nearest 0.1% *(m/m)* from the expression:

$$Q = \frac{\text{mass of solvent absorbed} \times 100}{\text{density of solvent} \times \text{mass of dried concrete sample}}$$

9.5 Determination of combined water and cement content

9.5.1 Sample preparation

9.5.1.1 Remove the slice from the polyethylene bag.

9.5.1.2 Heat the sample to constant mass, i.e. successive weighings after heating and cooling do not differ by more than 0.1 g, at (105 ±5) °C.

9.5.1.3 Treat the slice in accordance with **5.5.2** to produce a representative powdered sample of not less than 20 g all passing a 125 µm sieve and determine the combined water and cement contents of this powdered material in accordance with **9.5.2** to **9.5.5**.

9.5.2 Determination of combined water content

9.5.2.1 To determine the blank of the apparatus, carry out the procedure in **9.5.2.2** to **9.5.2.9** with no sample in the combustion boat. If the blank is greater than 1 mg, check the joints and ensure that they are free of leaks and repeat the blank determination. If a value greater than 1 mg is still obtained, replace the absorbents and repeat the procedure until a blank of less than 1 mg is obtained.

9.5.2.2 Using the apparatus detailed in **9.3.3**, determine the combined water content directly by igniting the powdered sample at (1 000 ±25) °C in a stream of dried air or nitrogen and weighing the evolved water after absorption on dried magnesium perchlorate.

NOTE This determination is only made when reliable control samples of the aggregates are available.

9.5.2.3 Purge the apparatus of residual moisture by passing dried gas through the combustion tube, heated to 1 000 °C, for 30 min with an empty absorption tube in position for the first 15 min, replacing it by the filled tubes for the rest of the time.

9.5.2.4 After 30 min, remove the source of heat and continue the air flow for a further 15 min. Remove the absorption tubes from the apparatus and transfer to the balance case.

9.5.2.5 When the combustion tube has cooled to below 100 °C, weigh the absorption tubes and fit into the train.

9.5.2.6 Weigh approximately 1 g of sample into a combustion boat previously ignited to constant mass at 1 000 °C and immediately insert into the combustion tube from the air inlet end.

9.5.2.7 Replace the connection and adjust the gas flow to about 3 bubbles per second at the bubbler.

9.5.2.8 Heat, cautiously at first, and maintain the temperature at 1 000 °C for 30 min. Remove the source of heat and continue to pass the gas for a further 15 min.

9.5.2.9 Disconnect the absorption tubes, and transfer to the balance case. Weigh after 15 min.

9.5.2.10 Calculate the combined water content, *X*, as a percentage to the nearest 0.1% (*m*/*m*) from the expression:

$$X = \frac{\text{gain in mass of absorption tubes} - \text{blank}}{\text{mass of sample}} \times 100$$

9.5.3 Determination of cement content

Determine the cement content of the prepared sample by the same method used for the analytical sample (see **6.9**).

9.5.4 Determination of aggregate porosity

9.5.4.1 Weigh approximately 1 kg of the dried coarse aggregate and determine the capillary porosity in accordance with **9.4**, but calculating it as a percentage, *q*.

9.5.4.2 Enclose the aggregate in a plastic mesh bag or support it on a sieve.

9.5.4.3 Dry the surface of the aggregate before reweighing by shaking off the excess liquid and rapidly rubbing in a cloth.

9.5.5 Determination of aggregate combined water content

9.5.5.1 Heat the solvent-saturated aggregate to constant mass (as in **9.5.1**) at (105 ±5) °C and crush to pass a 125 µm sieve.

9.5.5.2 Determine the combined water content in accordance with **9.5.2** but calculating it as a percentage, *Y*.

9.6 Calculation of the original water/cement ratio

9.6.1 Aggregate control samples available

In the absence of contrary information, assume that the values for porosity and combined water content found for the coarse aggregate are valid for the whole aggregate content of the sample.

Assuming that none of the water of hydration has been replaced by carbon dioxide, then:

a) original free water content = corrected capillary porosity + corrected combined water content, or:

$$W_\mathrm{f} = \left(Q - \frac{qF}{100}\right) + \left(X - \frac{YF}{100}\right) (\text{in } \%)$$

$$W_\mathrm{f} = Q + X - \frac{F}{100}(q + Y) (\text{in } \%)$$

original free water/cement ratio = $\frac{W_f}{C_1}$

b) original total water content = uncorrected capillary porosity + corrected combined water content, or:

$$W_t = Q + \left(X - \frac{YF}{100} \right) (\text{in } \%)$$

$$W_t = Q + X - \frac{YF}{100} \, (\text{in } \%)$$

original total water/cement ratio = $\frac{W_t}{C_1}$

where:

Q is the capillary porosity of the sample (in %) (see **9.4**);

q is the capillary porosity of the aggregate (in %) (see **9.5.4**);

X is the combined water content of the sample (in %) (see **9.5.2**);

Y is the combined water content of the aggregate (in %) (see **9.5.5**);

C_1 is the cement content of the concrete (in %) (see **9.5.3**);

F is the aggregate content of the concrete (in %) (see **6.9**).

9.6.2 Aggregate control samples unavailable

Assume the capillary porosity of the aggregate, q', (in %) has the same value as its water absorption, then the corrected capillary porosity is:

$$Q = \frac{q'F}{100} \, (\text{in } \%)$$

and the combined water content of the concrete is $0.23C_1$ so that:

the original free water/cement ratio = $\frac{Q}{C_1} - \frac{q'F}{100\,C_1} + 0.23$

NOTE If q′ is not reliably known then only the original total water/cement ratio may be reported.

The original total water/cement ratio = $\frac{Q}{C_1} + 0.23$

10 Type of cement

*COMMENTARY ON CLAUSE **10***
Differentiation between the various types of cement/addition that might have been used in a concrete presents problems of varying complexity. In favourable circumstances, it might be possible to differentiate between various cements by analysing for an element or elements peculiar to the cement/addition in question after careful separation of the cement matrix. For typical analyses of various types of cement see Annex B.

It is strongly advisable to supplement results thus obtained by examination under a microscope of a polished specimen containing cement particles still unhydrated.

10.1 General

Do not use this test to determine whether or not the cement used in the concrete conformed to a cement specification.

NOTE The test might help to determine conformity to or otherwise with any requirement referring to general cement type in a concrete specification.

10.2 Separation and analysis of matrix

10.2.1 Carefully break a solid piece of the concrete sample in, for example, a compressive strength testing machine, and remove, by sieving, material as fine as is possible, using, for example, a 63 µm sieve. If insufficient sample is obtained, further very careful breakdown is necessary.

10.2.2 Analyse the very fine material for insoluble residue (see **6.5.2**) and loss on ignition (see **6.6.6**) and, for example, soluble silica (see **6.5.3**), calcium oxide (see **6.5.4**), alumina, ferrous oxide, magnesium oxide, sulfuric anhydride, etc.

10.2.3 Assume that the insoluble residue represents aggregate in the fine material and the loss on ignition represents hydration and carbonation of the cement and correct the other analytical results by multiplying them by:

$$\frac{100}{100-(\text{loss}+\text{insoluble})}$$

10.2.4 Compare the results with typical analyses of various types of cement (see Annex B).

NOTE Any aggregate material in the separated fines can also contribute oxides, particularly ferrous oxide, alumina and silica.

10.3 Examination under a microscope

*COMMENTARY ON **10.3***
This examination should be made by a microscopist with experience of cement and concrete materials.

The test is usually carried out to establish whether CEM I cement or sulfate-resisting Portland cement has been used in the concrete. To do this, examine at least 10 anhydrous relics of cement grains greater than 40 µm in size, or at least 20 grains greater than 20 µm in size.

In older concretes which have been kept wet, e.g. foundations, it might prove impossible to find sufficient anhydrous grains.

It is often possible to determine whether either blastfurnace slag or fly ash is present. However, these identifications sometimes require the additional examination of thin sections in transmitted light and such a procedure is outside the scope of this part of BS 1881.

10.3.1 Reagents

10.3.1.1 *Mounting resin*, an epoxy or polyester resin that sets rigidly when mixed with an appropriate hardener.

10.3.1.2 *Silicon carbide powder*, medium grade, e.g. 26 µm.

10.3.1.3 *Non-aqueous liquid,* e.g. paraffin.

10.3.1.4 *Diamond paste,* of grades 14 µm, 6 µm, 3 µm, 1 µm and 0.25 µm.

10.3.1.5 *Non-aqueous solvent,* e.g. acetone.

10.3.1.6 *Potassium hydroxide solution, 10%.* Dissolve 10 g of potassium hydroxide in 100 mL of water.

10.3.1.7 *Methylated spirits or ethanol.*

10.3.1.8 *Hydrofluoric acid*, at 60% concentration.

10.3.2 Apparatus

10.3.2.1 *Rotary lap-plate*, using a non-aqueous polishing medium and diamond pastes.

10.3.2.2 *Reflected light microscope*, using magnifications up to ×800.

10.3.2.3 *Cylindrical mould,* at least 25 mm in diameter, e.g. a plastic tube.

10.3.3 Preparation of the sample

10.3.3.1 Use sample portion B obtained in accordance with **5.5.3**.

10.3.3.2 Break up the concrete sample and select at least 10 nominal 5 mm sized pieces of cement-rich-matrix as the test portion.

10.3.3.3 Dry the test portion at a temperature not exceeding 105 °C, allow it to cool and then embed the pieces in a low viscosity epoxy or polyester resin (**10.3.1.1**) by casting under vacuum into a cylindrical mould (**10.3.2.3**) and curing at the temperature recommended by the resin manufacturer.

NOTE A cylindrical cast specimen of approximately 25 mm diameter is usually found to be suitable but larger or multiple specimens are also acceptable.

10.3.3.4 Section the cylindrical casting with a diamond saw lubricated with a non-aqueous liquid (**10.3.1.3**) so that cut surfaces of the pieces are exposed on the sawn surface, and wash immediately using a non-aqueous solvent, e.g. acetone (10.3.1.5)[3].

10.3.3.5 Grind the sawn surface with silicon carbide powder (**10.3.1.2**) lubricated with a non-aqueous liquid such as paraffin, or other suitable oil (**10.3.1.3**), to remove saw marks.

10.3.3.6 Remove all traces of grinding powder.

NOTE Treatment with paraffin or industrial methylated spirits in an ultrasonic bath is effective.

10.3.3.7 Carefully polish with progressively finer diamond compounds (**10.3.1.4**) in an oil soluble base on a rotary lap-plate (**10.3.2.1**) with a suitable surface.

10.3.3.8 After a final polish with 0.25 µm diamond compound, clean the surface using a non-aqueous cleaning agent and dry with tissue. Adjust the grinding and polishing routine, particularly in terms of speed and duration of lapping, such that the plucking of cement particles out of the polished surface is minimized.

NOTE Control samples of known cement types should be prepared at the same time so that the polishing, etching and microscopical examination stages can be monitored.

10.4 Cement grain identification

10.4.1 Examine the polished surface through a reflected-light microscope (**10.3.2.2**) using magnifications up to ×800. If insufficient grains of anhydrous cement are found (see commentary on **10.3**), either prepare further polished samples as in **10.3.3** so that the required number of grains can be assessed, or report that the method is not applicable.

NOTE 1 Anhydrous relics of cement grains smaller than 20 µm may be examined in addition, but the mineralogical composition of such small particles might not be representative.

NOTE 2 Anhydrous relics of cement grains apparently exhibiting selective hydration throughout the grains, e.g. patches of hydrated matrix, should be discounted.

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[3] Prolonged immersion in acetone can soften epoxy and polyester resins.

10.4.2 Use chemical etching of the grains to reveal phases characteristic of the type of cement by employing one of the following procedures.

Immerse the polished surface in potassium hydroxide solution (**10.3.1.6**) at 30 °C for between 10 s and 20 s and wash it immediately in industrial methylated spirits or ethanol (**10.3.1.7**). Control the etching to give a bluish-grey colour to the tricalcium aluminate; the silicates remain grey, and the ferrite appears white.

Expose the polished surface to hydrofluoric acid vapour (**10.3.1.8**) for between 2 s and 5 s until the hexagonal alite is coloured straw-brown.

NOTE 1 The rounded belite appears in a variety of colours from blue to pink. In the interstitial phase, the tricalcium aluminate appears light grey and the ferrite appears white.

NOTE 2 It is sometimes difficult to distinguish these two compounds particularly at low magnification, i.e. less than ×500.

WARNING. Hydrofluoric acid is extremely dangerous and great care is required in its use, even in small quantities. Perform hydrofluoric acid vapour etching in a fume cupboard following the HSE leaflet *Hydrofluoric acid poisoning* [2] for the handling of this substance. The vapour can damage microscope lenses. Allow excess vapour to disperse completely from the specimen surface before examination is commenced. The vapour can be removed more quickly by placing a glass slide on top of the specimen after etching.

10.4.3 Using the compositional information given in Annex C, and making reference to the control specimens if prepared, identify the apparent type of cement represented by each anhydrous grain examined. Determine whether or not the cement type present in the concrete sample is that represented by at least 80% of the anhydrous grains examined.

10.4.4 If the dominant cement type represents fewer than 80% of the grains examined, repeat the procedure (**10.4.1** to **10.4.3**) and consider the combined findings. If the dominant cement type represents fewer than 80% of the grains examined overall, report the cement type as uncertain.

NOTE A mixture of cement types could be one explanation for such uncertainty, but mixtures are extremely unusual and such an interpretation should not be regarded as conclusive.

11 Type of aggregate

*COMMENTARY ON CLAUSE **11***
The identification of the type of aggregate is intended to aid the interpretation of the results of the chemical analysis. If more precise identification and classification of the aggregates are required, refer to a petrographer. The petrographical procedures required are outside the scope of this part of BS 1881.

Guidance notes for the petrographic examination of hardened concrete have been published as ASTM C856 [3] and Concrete Society Technical Report 71 [4].

Prepare a broken, or preferably a sawn, surface of the sample (see **5.5.3**). Identify the general character of the aggregate and compare the aggregate exposed with known samples whenever possible.

NOTE 1 Most carbonate aggregates can be identified by treating with dilute hydrochloric acid (1 + 9) and observing the presence (or absence) of effervescence. Even when a carbonate aggregate was not used in the concrete, there might be some carbonate particles, or particles containing carbonate which react with dilute acid, e.g. shell debris. Some dolomites do not react rapidly with cold dilute hydrochloric acid but effervesce with warm dilute hydrochloric acid.

NOTE 2 Examination of the aggregates and of their reaction with acids can be aided by observation under a low-power binocular microscope.

12 Other constituents

*COMMENTARY ON CLAUSE **12***
*The analytical sample prepared in accordance with **5.5.2** may be used for the determinations of chloride, sulfate and alkali contents. However, the test for alkali content might require a sample of separated fines, to avoid aggregate interference (see **12.3**). Since the chloride and sulfate contents are usually reported in relation to the cement content of the concrete, such a sample, in which the cement matrix has been concentrated, might present some advantages in all these tests.*

12.1 Determination of chloride content

12.1.1 Preparation of separated sample

Break the concrete sample and remove fine material by sieving through a 125 µm sieve until a sufficient quantity of fines has been produced. The less the aggregate is damaged the higher the cement content of the fines. Determine the cement content of this separated sample using the procedure described in **6.9**.

NOTE 1 Alternative methods based on potentiometric titration are acceptable.

*NOTE 2 Salts of thiocyanates and thiosulfates are increasingly used as accelerating admixtures in place of chlorides. These compounds can interfere with the determination of chloride content. Hence, a short period of boiling after adding nitric acid to the sample has been introduced into the procedure in **12.1.3** to decompose these compounds. Quantitative methods for thiocyanates and thiosulfates are being developed.*

12.1.2 Reagents

*NOTE See **6.2.1**.*

12.1.2.1 *Nitric acid,* relative density 1.4.

12.1.2.2 *Silver nitrate standard solution, 0.1 mol/L.* Dry powdered silver nitrate at 150 °C for 2 h. Cool in a desiccator and dissolve 16.989 g in water and dilute to 1 L. Store the solution in an opaque glass bottle and protect from prolonged exposure to light.

12.1.2.3 *Thiocyanate standard solution, approx. 0.1 mol/L.* Dissolve 7.6 g of ammonium thiocyanate or 9.7 g of potassium thiocyanate in water and dilute to 1 L. Standardize against the silver nitrate standard solution using iron III indicator, once a week or each time a determination of chloride content is made, if less frequent.

12.1.2.4 *Iron III indicator solution.* To 50 g ammonium ferric sulfate add 60 mL of water and warm to dissolve. Add 10 mL of nitric acid, cool, and store in a glass bottle.

12.1.2.5 *3,5,5-trimethylhexanol (nonyl alcohol).*

12.1.3 Procedure

12.1.3.1 Weigh into a stoppered 500 mL conical flask (5 ±0.005) g of the analytical (see **5.5.2**) or separated (see **12.1.1**) sample.

12.1.3.2 Disperse with 50 mL of water and add 10 mL of nitric acid (**12.1.2.1**). Add 50 mL of hot water, boil for between 4 min and 5 min and keep warm for between 10 min and 15 min. If the supernatant liquid is turbid, filter through a fast hardened ashless filter paper (see **6.3.1**, Note) and wash with hot water.

12.1.3.3 Cool to room temperature and add a measured excess of silver nitrate standard solution (**12.1.2.2**).

12.1.3.4 Add 2 mL to 3 mL of 3,5,5-trimethylhexanol (**12.1.2.5**), stopper the flask and shake vigorously to coagulate the precipitate. Add 1 mL of iron III indicator solution (**12.1.2.4**) and titrate with thiocyanate standard solution (**12.1.2.3**) to the first permanent red colour.

12.1.3.5 Calculate the chloride ion content, J, as a percentage of the cement to the nearest 0.01% (m/m) from the expression:

$$J = \left(V_5 - \frac{V_6 m}{0.1} \right) \frac{0.354\,5}{M_\mathrm{c}} \times \frac{100}{C_1}$$

where:

M_c is the mass of sample used (in g);

V_5 is the volume of 0.1M silver nitrate solution added (in mL);

V_6 is the volume of thiocyanate solution used (in mL);

m is the molarity of the thiocyanate solution (in *mol/L*);

C_1 is the cement content of the sample used (in %).

NOTE Other concentrations of silver nitrate solution and thiocyanate solution may be used and the equation modified accordingly.

12.2 Determination of sulfate content

12.2.1 Reagents

12.2.1.1 *Hydrochloric acid*, relative density 1.18.

12.2.1.2 *Dilute hydrochloric acid (1 + 49)*.

12.2.1.3 *Ammonium hydroxide solution (1 + 1)*.

12.2.1.4 *Barium chloride solution, 100* g/L, obtained by dissolving 100 g of barium chloride in 1 L water.

12.2.1.5 *Methyl red indicator solution*, obtained by dissolving 1 g of methyl red in 600 mL of methylated spirits and add 400 mL of water.

12.2.2 Procedure

12.2.2.1 Weigh into a 400 mL beaker (5 ±0.005) g of the analytical (see **5.5.2**) or separated (see **12.1.1**) sample.

12.2.2.2 Disperse with 50 mL of water and add 10 mL of concentrated hydrochloric acid (**12.2.1.1**).

12.2.2.3 If effervescence is considerable, immediately cover the beaker. Add 50 mL of hot water, cover the beaker and boil the solution gently for 5 min to 10 min.

12.2.2.4 Filter through a medium ashless filter paper (see **6.3.1**, Note), washing the residue thoroughly with hot dilute hydrochloric acid (1 + 49) (**12.2.1.2**).

12.2.2.5 Add three drops of the methyl red indicator (**12.2.1.5**) and heat the filtrate to boiling. If necessary, just neutralize to yellow with dilute ammonium hydroxide solution (**12.2.1.3**).

12.2.2.6 Immediately add 1 mL of concentrated hydrochloric acid (**12.2.1.1**) and then add dropwise to the boiling solution 10 mL of barium chloride solution (**12.2.1.4**).

12.2.2.7 If excess ammonium hydroxide was added, 1 mL of concentrated hydrochloric acid might not be sufficient to obtain the required acid solution and the barium sulfate precipitate will then be contaminated. In this case repeat the test.

12.2.2.8 Boil gently for 5 min, keep the solution at just below boiling for 30 min and allow to stand at room temperature for 12 h to 24 h.

12.2.2.9 Filter through a slow ashless filter paper (see **6.3.1**, Note) and wash free from chlorides with hot water.

12.2.2.10 Transfer the paper and contents to a weighed silica or platinum crucible and burn off the paper without flaming. Ignite the precipitate at 800 °C to 900 °C until constant mass is achieved (see **6.6**).

12.2.2.11 Calculate the sulfate content, *G*, expressed as SO_3, as a percentage of the cement to the nearest 0.1% (*m/m*) from the expression:

NOTE SO_4 is the correct symbol for sulfate but engineers conventionally report sulfate as SO_3.

$$G = \frac{L}{M_\mathrm{d}} \times 34.3 \times \frac{100}{C_1}$$

where:

M_d is the mass of the sample used (in g);

C_1 is the cement content of the sample used (in %);

L is the mass of ignited barium sulfate (in g).

*NOTE The stock test solution (see **6.5.3**) may be used for this determination. Use a 200 mL aliquot and proceed as before from the heating stage to boiling and neutralizing with dilute ammonium hydroxide solution. In the calculation, M_d is 2 g.*

12.3 Determination of sodium oxide and potassium oxide contents

*NOTE Many aggregates contain sodium and potassium compounds which are not readily available for reaction in concretes. The method of analysis given in this subclause could, however, extract the alkalis from a finely ground sample of such aggregate. When a result with minimum interference from aggregate is required, the test should be made on a sample of separated fines (see **12.1.1**) and care taken to minimize contamination of the fines with aggregate.*

12.3.1 Apparatus

12.3.1.1 *Flame photometer*, conforming to BS EN 196-2:2013, **4.3.21**.

12.3.2 Reagents

12.3.2.1 *Solutions for calibrating the flame photometer*, as specified in BS EN 196-2:2013, Table 4.

12.3.2.2 *Concentrated hydrochloric acid*, as specified in BS EN 196-2:2013, **4.2.1**.

12.3.3 Calibration

Calibrate the flame photometer in accordance with BS EN 196-2:2013, **4.5.19.3**.

12.3.4 Procedure

12.3.4.1 Weigh into a 400 mL beaker, made from a material that does not yield sodium or potassium to the solution, (5 ±0.005) g of the analytical sample (see **5.5.2**) or (2 ±0.005) g of the separated sample (see **12.2.1**), disperse with 150 mL of water then add 20 mL of concentrated hydrochloric acid (**12.3.2.2**) and 50 mL phosphoric acid (1+19).

12.3.4.2 Boil the solution gently for 10 min, cool and quantitatively transfer to a 500 mL volumetric flask.

12.3.4.3 Dilute to the mark with water, mix thoroughly and filter off about 100 mL through a dry medium ashless filter paper (see **6.3.1**, Note).

12.3.4.4 Pipette 25 mL of the filtrate into a 100 mL volumetric flask, dilute to the mark with cold water and mix thoroughly before spraying onto the flame.

12.3.4.5 If the sample gives a reading greater than given by the S7 solution for either sodium or potassium, pipette 25 mL of the filtrate into a 200 mL graduated flask dilute to the mark with cold water, mix thoroughly and respray.

12.4 Calculation

From the respective calibration graphs for sodium oxide and potassium oxide (see **12.3.3**) convert flame photometer readings to concentrations (in mg/L).

Calculate the sodium oxide and potassium oxide contents as percentages to the nearest 0.01% (*m*/*m*) of the sample used from the expression:

$$H = \frac{0.2k}{M_n}$$

where:

H is the alkali oxide content (in %);

k is the concentration of alkali (in mg/L);

M_n is the mass of the sample (in g).

Where the sample gave a reading greater than given by the S7 solution for either sodium or potassium and filtrate was pipetted into a 200 mL graduated flask:

$$H = \frac{0.4k}{M_n}$$

Then calculate the sodium oxide equivalent, N_e, as a percentage (*m*/*m*) of the cement from the expression:

$$N_e = \frac{[u + (w \times 0.658)] \times 100}{C_1}$$

where:

C_1 is the cement content of the sample used (in %);

u is the sodium oxide content (in %);

w is the potassium oxide content (in %).

*NOTE The calculated value of N_e can be higher than the true alkali content of the cement used due to contributions from sources other than the cement, particularly when the analytical sample (see **5.5.2**) has been used for analysis.*

The sodium oxide equivalent content of the concrete, N_b, is calculated in kg/m^3 from:

a) where the analytical sample (see **5.5.2**) was used:

$$N_b = \frac{[u + (w \times 0.658)]}{100} \times \rho_9$$

where:

u is the sodium oxide content (in %);

w is the potassium oxide content (in %);

ρ_9 is the oven dried density of concrete (in kg/m^3)

or:

where a sample of separated fines (see **12.1.1**) was used:

$$N_b = \frac{N_e}{100} \times \frac{C_1}{100} \times \rho_9$$

where:

C_1 is the cement content (m/m) of the concrete, see **6.9** (in %).

13 Report

13.1 General

State in the report that the analysis was carried out in accordance with this part of BS 1881. When alternative methods are used (see Commentary on Clause **6**), include details of them and evidence that they are of equivalent accuracy.

13.2 Information to be included in the report

The report shall include:

a) date and place of sampling and identification marks and other relevant details supplied with the sample;

b) full qualitative description of the sample, including the type of aggregate, with particular reference to factors likely to reduce the accuracy of the results;

c) date and place of the analysis;

d) results of the determinations as mean values;

e) any assumptions made in the analysis, e.g. type of aggregate, cement analysis, aggregate analysis, etc.; and

f) contents of the constituents derived from the calculations with the magnitude of probable errors.

NOTE The report may include:

a) an interpretation of the results of the analysis in accordance with the information requested, stating specifically the method used to determine the cement content;

b) any other results obtained coincidentally to the tests required;

c) the results of any additional tests carried out at the analyst's discretion; and

d) conclusions regarding the concrete quality where required.

Annex A (informative) Precision

Estimates of the repeatability and reproducibility of hardened concrete analysis for cement content are given in Table A.1. The data have been obtained for concretes made with CEM I cement and might not be applicable to other types of cement.

The precision data were determined from an experiment conducted in 1983. Each of 18 laboratories analysed split level pairs of samples of three different concretes. The results from two of the laboratories were rejected as outliers using the criteria of BS ISO 5725-2. If the outlier results had been included in the statistical analysis the effect would have been to increase repeatability, *r*, by about 50% and reproducibility, *R*, by about 100%. In Table A.1, σ_r is the standard deviation of the within-laboratory component of precision and σ_L is the standard deviation of the between-laboratory component as described in BS ISO 5725-2.

The difference between two single results found on identical samples of the same concrete by one analyst within a short time interval exceeds the repeatability, *r*, on average not more than once in 20 cases in the normal and correct operation of the test method.

The difference between two single test results found on identical samples of the same concrete by two analysts working in different laboratories at different times exceeds the reproducibility, *R*, on average not more than once in 20 cases in the normal and correct operation of the test method.

In the definitions, a single test result is the cement content calculated from the average of duplicate analyses of one analytical sample.

Table A.1 **Precision estimates**

Type of aggregate	**Range of cement contents investigated** kg/m^3	**Oxide used to calculate cement content**	***r*** kg/m^3	***R*** kg/m^3	σ_r kg/m^3	σ_L kg/m^3
Flint	240 to 425	CaO	40	60	15	15
		Soluble SiO_2	40	60	15	15
Limestone	300 to 350	Soluble SiO_2	40	50	15	10

Annex B (informative)

Typical analyses of types of cement currently available in the UK

Table B.1 gives typical analyses of types of cement currently available in the UK.

Table B.1 **Typical analyses of types of cement currently available in the UK**

	Portland BS EN 197-1 CEM I	**Limestone BS EN 197-1 CEM II/AL or LL**	**Blastfurnace BS EN 197-1 CEM III** (see Note 2)	**Portland fly ash BS EN 197-1 CEM II/B-V** (see Notes 3 and 4)
	%	%	%	%
CaO	64.5	62	51	51
SiO_2	20.2	16	16	26 (16)
MgO	1.6	1	7	2
Al_2O_3	5.4	3	9	11
Fe_2O_3	2.6	5	2	5
SO_3	2.8	2	2	3

NOTE 1 These values are subject to a variation of at least ±10%, with the exception of CaO content in CEM I cement which is subject to a variation of approximately ±3%.

NOTE 2 Variations in the clinker and slag used and in their relative proportions may cause large deviations from these values.

NOTE 3 Variations in the clinker and fly ash used and in their relative proportions can cause large deviations from these values. The value in parenthesis for silica is that likely to be determined under the conditions of extraction used in concrete analysis. The conditions of extraction might not bring all the other oxides into solution from the pozzolana although it might be expected that all the calcium would dissolve.

NOTE 4 The soluble silica for Portland fly ash varies with age, as the soluble silica of fly ash changes in the presence of Portland cement

Annex C (informative)

Mineral phase compositions of different types of cement

Portland (CEM I) cements not having a specification limit for tricalcium aluminate content, e.g. CEM I conforming to BS EN 197-1, generally have a volume ratio of ferrite to tricalcium aluminate of less than 2:1. For sulfate-resisting Portland cement conforming to BS EN 197-1, the volume ratio of ferrite to tricalcium aluminate is generally more than 5:1. However, this ratio might not apply to sulfate-resisting Portland cements with a high silicate content, such that the total volume of ferrite and tricalcium aluminate is very low.

Up to 20% of the unhydrated cement grains examined, greater than 20 μm in size, can display an apparently anomalous phase composition. For this reason a concrete made with sulfate-resisting Portland cement conforming to BS EN 197-1, for example, might contain a significant number of grains showing larger than expected amounts of tricalcium aluminate and thus having the appearance of CEM I cement. Finding a few apparently anomalous grains in the examination is therefore not necessarily an indication of adulteration of the cement used.

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For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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