BS 1881 : Part 124 : 1988

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British Standard

Testing concrete

Part 124. Methods for analysis of hardened concrete

Essais du béton
Partie 124. Méthodes d'analyse du béton durci

Prüfung von Beton
Teil 124. Prüfung von Festbeton

Foreword

This Part of BS 1881 has been prepared under the direction of the Cement, Gypsum, Aggregates and Quarry Products Standards Committee. It supersedes BS 1881 : Part 6 : 1971, which is withdrawn.

The publication as BS 1661 : Part 6 of the first British Standard methods for analysing hardened concrete enabled analysts to use the same methods and to make the same assumptions. After 12 years' use some modifications and extensions to the methods and interpretation of results are necessary.

The publication in 1960 of the revision of BS 4551 also made it desirable that the methods for analysing hardened concrete should be in harmony with the methods for analysing hardened mortars and screeds.

The most important technical changes made in this revision as a result of experience gained with the methods in these two standards are as follows.

(a) Deletion of the sodium succinate step for removing silica, iron and alumina prior to measuring calcium; on occasion this led to low results probably caused by co-precipitation of calcium with the silica.

(b) A change in the method for extracting soluble silica; the new method has been shown to give reduced attack on siliceous aggregate while still giving full extraction of the soluble silica. This change in the extraction procedure renders the new method unsuitable for analysing concretes made with high alumina cement.

(c) Deletion of the method of cement content determination associated with the procedure for measuring aggregate grading since it was considered that few (if any) workers still used this procedure.

(d) Inclusion of a method for determining sulphide as an essential step in analysing concretes containing ground granulated blastfurnace slag as a reactive addition.

(e) Inclusion in the test for original water content of a sanction for assuming the combined water content of hydrated Portland cement in hardened concrete when samples of the aggregates are not available. It is recognized that making the assumption is usually more accurate than attempting to measure the combined water content of the sample and of the aggregate recovered from the concrete, and making the appropriate corrections.

(f) Simplification of the equations for calculating cement and aggregate contents based on an assumed value for the combined water content of hydrated Portland cement in hardened concrete.

(g) Deletion of the method for the determination of sulphoaluminate on account of doubts about its reliability and utility.

(h) Deletion of the method for the determination of cement content by calculation from the acid soluble calcium and carbon dioxide contents; the method frequently gave difficulties caused by uncertainty in the assumed value for carbonation of the concrete and was therefore considered unsuitable for regular use. However, in certain cases it allows an estimate of the cement content to be made when no other method seems possible.

In addition some minor changes have been made, among which is a new recommended indicator, calcein, for calcium determination because the indicator previously recommended, HSN, does not always give clear end-points, however, when clear unambiguous end-points are given, HSN may be preferred. Trichlorethane is now used for resaturation when measuring capillary porosity as the toxic nature of carbon tetrachloride has been recognized.

A universally applicable method of analysis does not exist owing to the wide variety of materials from which concrete can be made and the conditions to which it may have been subjected. Many different approaches are therefore necessary in varying circumstances and in this Part of BS 1681 all possible circumstances have not been included. Nevertheless the methods given are considered to present the best approach in the majority of cases.

The increasing use of reactive additions in concrete will complicate the analysis of concretes. The content of blastfurnace slag from known sources can still be determined, but at present no simple method exists for determining the content of pozzolanas, e.g. pulverised-fuel ash.

Sampling hardened concrete to provide sufficient data to give meaningful information about the composition of a batch of concrete remains a problem. No statistical statement about the results of analysis can be made with less than two independent samples, and this should be regarded as the minimum number.

It should not be necessary to stress that the results for one batch of concrete cannot be applied to any other batch.

Poor results are often caused by incorrect sample preparation and the requirements given in 4.5 should be followed closely. Precise and accurate results can be achieved using the methods described. This does not imply that all analysts will regularly achieve such results.

Contents

Methods

1 Scope

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

The procedures apply to concretes made with Portland cements and, in favourable circumstances, containing ground granulated blastfurnace slag.

The analysis of concretes made with other cements and the determination of pulverized-fuel ash content are outside the scope of this Part of BS 1881.

NOTE, The titles of the publications referred to in this standard are listed on page 21.

2 Definitions

For the purposes of this Part of BS 1881, the definitions in BS 5328 and BS 6106 : Part 6 apply.

3 Sampling

3.1 General

It is usual for the tests described in this Part of BS 1881 to be applied only when there is some doubt 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 may 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 will be 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 such reasons it is extremely difficult to specify how a sample should be taken and no requirements can be given.

NOTE. It is strongly advised that no sample he 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 3.3).

3.2 Selection of sample

Observe the following general requirements in selecting the concrete sample.

(a) Ensure that the minimum linear dimension of the sample is at least five times that of the nominal maximum dimension of the largest aggregate particles.

(b) Ensure that the sample is in a single piece, with no evidence of fracture cracking apparent, if original water content is to be determined.

(c) The minimum mass of the piece will be determined by testing requirements but a mass of. at least 1 kg is necessary, or at least 2 kg if the original water content is to be determined, or at least 4 kg if aggregate grading is to be determined.

(d) Avoid all foreign matter and reinforcement unless they are subject to test.

Clearly and indelibly label the sample with full particulars, including the date, exact position from which the sample was taken, the method of sampling and any other relevant information. Enclose the sample in a heavy-duty polyethylene bag and seal or tie it securely.

3.3 Number of samples

When information about the composition of concrete with a volume of up to 10 m^3 is required, take at least two and preferably four 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 at least ten independent samples and analyse them separately. The results can then be used to identify locations requiring more extensive investigation.

3.4 Additional material

In addition to the concrete samples, information on the wmposition of the cement and aggregate used in the concrete will improve the accuracy of the test results. Where available, take representative samples of all the constituents of the concrete used, in accordance with the methods given in the relevant British Standard where applicable, enclose them in heavy-duty polyethylene bags, clearly label and seal or tie securely. Where such samples are not available, give any information on the source and nature of these materials which will be helpful.

3.5 Certificate of sampling

Each sample of concrete and additional material shall be accompanied by a certificate from the person responsible for taking the sample stating that sampling was carried out in accordance with this Part of BS 1881. The certificate shall include the following:

(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 (4.3 requires the orientation of the sample to be known);

(g) the name of the sampler;

(h) the signature of the person responsible for sampling.

4 Treatment of samples

4.1 Apparatus

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

4.1.2 *Suitable crushing and grinding equipment.* Jaw crushers, hammer mills, disc grinders, gyratory mills and mechanical or hand-operated mortars and pestles are among the types which have proved to be satisfactory.

4.1.3 150 µm *test sieve,* complying with BS 410, complete with the appropriate size of lid and receiver. Similar test sieves with apertures of 5.0 mm, 2.36 mm and 600 pm will also be required if mechanical crushing and grinding is not used (see 4.5.2).

4.1 A *Permanent bar magnet.*

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

4.1.6 Saw, fitted with a diamond or Carborundum blade, when original water content is to be measured.

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

4.2 Density

When the content of constituents determined as percentages by mass of the oven-dried (105 °C) concrete is to be converted to kilograms per cubic metre of concrete, then before beginning any work involving breakdown of the concrete samples, measure the oven-dried (105 °C) density of a single sound piece of concrete. For this purpose, immerse the piece of concrete in water for at least 30 min, and then determine its volume (V_3) by water displacement and its oven-dried density (ρ_9) using the procedures in BS 1881 : Part 114.

43 Original water content

When the original water content is to be determined, cut a slice from the sample with two parallel cuts, preferably with a diamond saw (4.1.6), avoiding as far as possible loss of material out of the cut faces. The slice shall be approximately 20 mm thick and have a single face area of not less than 10 000 mm².

The direction of sawing shall be from top to bottom of the concrete as cast where this can be identified. With horizontally drilled cores from columns, walls etc., which are more than 1 m high it is permissible to cut the slice parallel to the axis of the core to provide a specimen of adequate size.

Samples which have a cross-sectional area of less than 2500 mm’, e.g. 50 mm cubes or 50 mm diameter cylinders or cores shall be tested for water content without sawing. The test shall then precede that for cement content. To reduce carbonation of the concrete, store specimens for water content determination in airtight containers until ready for testing.

4.4 Nature of aggregate

Examine the concrete sample (see 9.2), and broadly classify both coarse and fine aggregate under the following headings:

- type I: natural aggregates essentially insoluble in dilute hydrochloric acid;
- type S: natural aggregates largely soluble in dilute hydrochloric acid:
- type 0: other aggregates.

4.6 Preparation of test samples

4.5.1 *Initial treatment.* Break up the sample into lumps not larger than about 50 mm in size, avoiding as far as possible fracture of the aggregate. Dry the lumps in the oven (4.1.1) at 105 ± 5 °C for 15 h to 24 h and allow to cool to room temperature.

Divide the dried sample into two portions, A and B, so that portion A is approximately threequarters of the bulk and portion B is the remainder. Ensure as far as possible that each portion remains representative of the whole.

4.5.2 *Treatment of portion A.* A major source of error in the analysis of hardened concrete is inadequate sample preparation. It is essential to avoid loss of material, particularly dust, during the crushing and grinding operations. Avoid excessive particle size reduction during the preparation of the analytical sample. Carry out the crushing, grinding and following operations as quickly as possible so that the sample is exposed to atmospheric carbon dioxide for the minimum time. A suitable procedure to provide a representative sub-sample of not less than 20 g of ground material, all passing a 150 µm sieve, to be used as the analytical sample is as follows.

Using suitable crushing and grinding equipment (4.1.2), crush portion A until it all passes a 5.0 mm sieve (4.1.3). Using a riffle box or by equivalent means (4.1.7). subdivide this to produce a subsample of 500 g to 1000 g. Crush this sub-sample to pass a 2.36 mm sieve and reduce by two separate subdividing operations, discarding one half on each occasion. Crush the whole of the remaining part to pass a 600 µm sieve and again reduce by two separate subdividing operations, discarding one half on each occasion. Crush the whole of the remaining part to pass a 150 µm sieve.

When mechanical means of crushing and grinding are used some of these stages may be by-passed. For example, the initial crushing of portion A may yield a product passing a 1.18 mm sieve. In this case the subdivision before the next stage should provide a sub-sample of 100 g to 200 g. This can then all be ground in a suitable grinder to pass a 150 µm sieve.

NOTE. Before the final grinding operation, it is recommended that material in the sub-sample which passes a 150 µm sieve is removed by sieving, 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.

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 (4.1.4). Transfer the analytical sample to a clean dry bottle with an airtight closure (4.1.5) and thoroughly mix the sample by tumbling, rolling or shaking for at least 2 min.

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

4.5.3 Treatment of portion B. Retain portion B in an airtight bottle for use in the tests given in clauses 6, 8, 9, and 10.

5 Cement and aggregate content

5.1 General

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 5.9). Wherever possible, therefore, representative samples of the cement and aggregate used in making the concrete shall be obtained and chemical analyses carried out on them in a similar manner to that described for the concrete analytical sample.

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 appendix 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.

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 5.9.3 and appendix 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 about 110 kg/m^3.

Use one or more of the methods detailed in this clause as appropriate. In 5.5.1 a method for the extraction of soluble silica is described, and it is essential that this method is followed exactly. Methods of proven accuracy are given for other determinations. It is permissible to use alternative methods for the determination of the following:

(a) calcium oxide (see 5.4), e.g. by atomic absorption;

(b) soluble silica (see 5.5.3). e.g. by atomic absorption;

(c) loss on ignition (see 5.6). e.g. by thermogravimetry;

(d) carbon dioxide (see *5.71,* e.g. by instrumental methods;

(e) sulphide (see 5.8). e.g. by instrumental methods;

provided evidence is presented that such methods give equivalent results.

Duplicate analyses shall be made unless a series of concrete samples from a single batch is being tested. Duplication shall preferably be carried out from the sample preparation stage, e.g. two analytical samples (4.5.1) are to be prepared and each analysed separately.

5.2 Reagents

5.2.1 *General.* All reagents shall be of analytical reagent quality, and distilled or deionized water shall be used throughout the analysis.

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 of solid reagents if the solution is not dear. Prepare the reagents by the following methods.

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

522 Ammonium chloride solution, 1 g/L. Dissolve 1 g of ammonium chloride in 1 L of water.

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

5.24 Calcium standard solution, approximately 1.00 g CaO/L. Weigh 1.78 g to 1.79 g of pure calcium carbonate dried at a temperature in the range 150 °C to 208 °C and record the mass to the nearest 0.0001 g. Dissolve in a slight excess of dilute hydrochloric acid (1+4), boil to expel carbon dioxide, cool and dilute to 1 L in a volumetric flask.

Express the concentration of CaO in g/L to three significant figures as mass $CaCO_3$/1.7848.

5.2.5 Triethanolamine solution (1+4).

5.2.6 Sodium hydroxide solution, 200 g/L. Dissolve 200 g of sodium hydroxide in 1 L of water.

5.2.7 EDTA standard solution, Dissolve 6.67 g of EDTA (ethylenediamine-tetra-acetic acid, disodium dihydrate salt) in warm water, filter if necessary, cool and dilute to 1 L. Store in a polyethylene bottle. Standardire against standard calcium solution as follows.

Pipette 20 mL of the calcium standard solution (5.2.4) into a 250 mL beaker or flask, add 10 mL of triethanolamine solution (5.2.5) and 10 mL of sodium hydroxide solution (5.2.6) and dilute to about 150 mL. Add about 0.15 g calcein indicator *(5.23)* and titrate with the EDTA solution until the fluorescent green of the indicator completely changes to pink-purple with no residual fluorescence. The end-point is more easily seen if observed against a black background. Calculate the CaO equivalent of the EDTA, *E,* (in mg CaO/mL) from the expression:

$$E = \frac{20 \times h}{t}$$

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.

5.2.8 *Hydrochloric acid,* relative density 1.18.

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

5.210 *Polyacrylamide solution,* 1 g/L. Add 0.1 g of polyacrylamide, molecular weight about 5 x 10^6, to 100 mL of water. Stir using a mechanical stirrer until dissolved (see note 1 to 5.5.1).

52.11 *Polyethylene oxide solution, 2.5* g/L. Slowly add 0.5 g of polyethylene oxide, molecular weight less than 1 x 10^6, to 200 mL of water, stirring using a mechanical stirrer until dissolved. Discard after two weeks.

5.2.12 *Sodium carbonate solution,* 50 g/L. Dissolve 50 g of anhydrous sodium carbonate in 1 L of water.

5.2.13 *Orthophosphoric acid,* relative density 1.7, used when carbon dioxide is to be determined.

5.2.14 *Starch solution,* used when sulphur as sulphide is to be determined. 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.

5.2.15 *Potassium iodate standard solution,* used when sulphur as sulphide is to be determined. Dry pure potassium iodate at 120 ± 5 °C, and allow to cool in a desiccator. Weigh out 1.112 g to 1.113 g and dissolve with 12 g of potassium iodide and two pellets of sodium hydroxide in freshly boiled and cooled water. Dilute the solution to 1000 mL in a volumetric flask, mix and store in a glass-stoppered bottle. 1 mL of this solution is equivalent to 0.5 mg sulphide. This potassium iodate solution is stable for several months.

5.2.16 *Sodium thiowlphate wlution,* used when sulphur as sulphide is to be determined. Dissolve 7.4 g of sodium thiosulphate ($Na_2S_2O_3 .5H_2O$) in water and make up to 1 L. Standardize this solution by the following method. Pipette 20 mL of the potassium iodate standard solution (5.2.15) into a 500 mL beaker or flask, dilute to approximately 300 mL with water and add 40 mL of dilute hydrochloric acid (1 + 1) (5.2.9). Immediately titrate with the sodium thiosulphate solution until the colour of the liquid becomes a pale yellow and then add 2 mL of the starch solution (5.2.14). Continue the titration until the colour changes from blue to colourless. Record the volume, *V,* of sodium thiosulphate solution required to the nearest 0.01 mL.

Calculate the factor, *T,* of the sodium thiosulphate soiution from the expression:

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

52.17 *Stannous chloride, solid,* used when sulphur as sulphide is to be determined ($SnCl_2 .2H_2O$).

5.2.18 *Ammoniacal zinc sulphate wlution, used* when sulphur as sulphide is to be determined. Dissolve 50 g of zinc sulphate ($ZnSO_4 .7H_2O$) in 150 mL of water and 350 mL of concentrated ammonium hydroxide solution. Allow to stand for at least 24 h and filter.

5.3 Apparatus

5.3.1 *General.* All volumetric glassware shall have an accuracy of class B or better as given in the appropriate British Standard.

NOTE. It is assumed that a laboratory carrying out these tests will be equipped with basic apparatus such es analytical balances, beakers, volumetric flasks, pipettes, burettes, filtretion apparatus, magnetic end mechanical stirrers, etc. Only apperatus special to the determinations necessary to analyse hardened concrete is therefore listed. The filter papers* used should be ashless.

53.2 250 mL *polypropylene beakers.*

53.3 Plastics stirring rods.

5.3.4 *Furnace,* or furnaces controlled to maintain temperatures of 925 ± 25 °C and 1200 ± 50 °C.

5.3.5 Boiling water or steam bath.

53.8 Desiccator, containing dried magnesium perchlorate.

5.3.7 Apparatus for the determination of carbon dioxide. The apparatus consists of 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 sulphuric acid and then through a series of absorption tubes which successively contain pumice coated with anhydrous copper sulphate, 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. A typical apparatus is shown in figure 1. The apparatus shall be free from leaks.

5.3.8 Apparatus for the determination of sulphur as sulphide. The neck of a 250 mL round bottom flask shall be fitted with a groundjoint assembly, providing a means of adding acid and of passing air or other inert gas through the solution in the flask, an antisplash device or a condenser and a delivery tube for the issuing gas. The flexible connecting tubes shall be made of sulphur-free material, polyvinyl chloride or silicone rubber. The delivery tube dips into the solution in a 490 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 sulphide or sulphur dioxide. A typical apparatus is shown in figure 2.

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*For information on the availability of filter papers, apply to Enquiry Section. BSI, Linford Wood, Milton Keynes MK14 6LE. enclosing a stamped addressed envelope for reply.

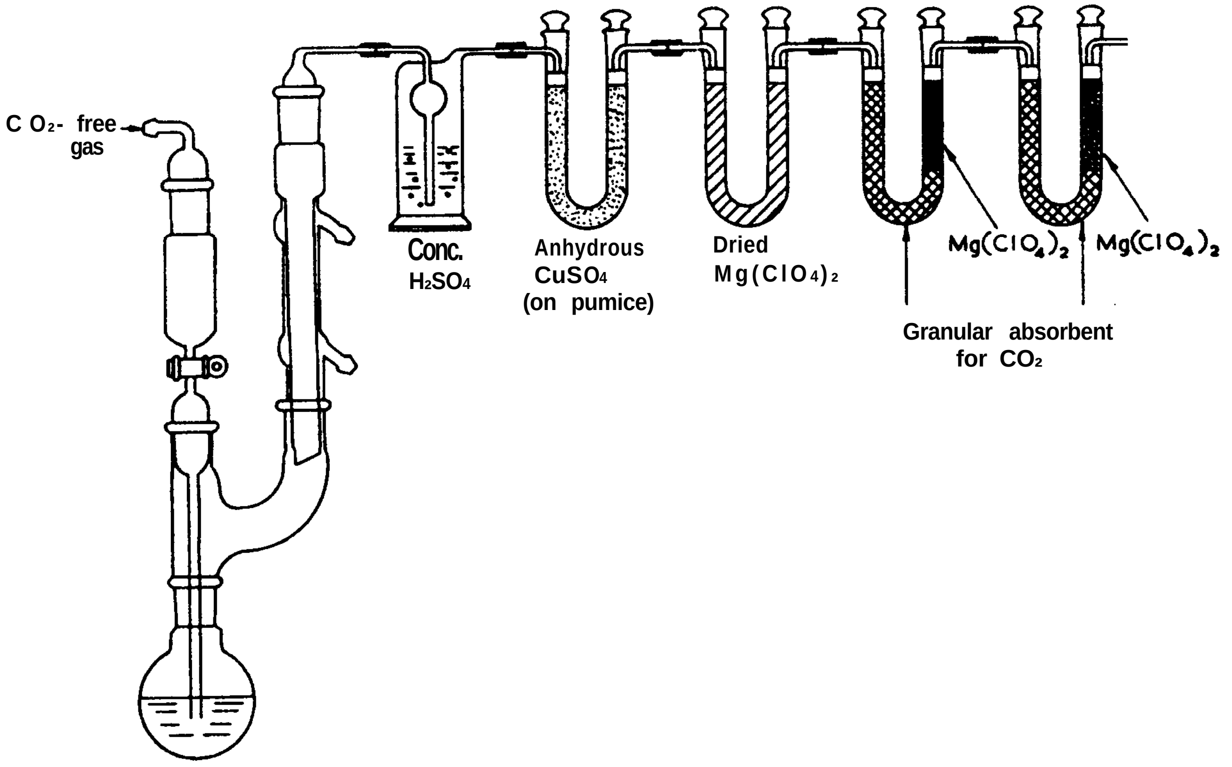


Figure 1. Typical apparatus for determination of carbon dioxide content

5.4 Determination of calcium oxide when insolubk residue and soluble silica are not also to be determined

Weigh 5 ± 0.005 g of the analytical sample as prepared in 4.5.2 and transfer into a 500 mL beaker. Disperse with 50 mL to 100 mL of cold water, add 10mL of concentrated hydrochloric acid (5.2.8), swirl, immediately add hot water to about 150 mL, bring to the boil and keep hot for 5 min. After cooling, transfer the solution quantitatively to a 500 mL volumetric flask, dilute to the mark with water and mix thoroughly. Filter a portion of the solution through a dry medium ashless filter paper (see note to 53.1) or, alternatively, use a centrifuge to obtain a dear solution.

Pipette a 25 mL aliquot of the clear solution into a 250 mL beaker or flask and dilute to about 150 mL. Add 10 mL of the triethanolamine solution, 10 mL of the sodium hydroxide solution and about 0.15 g of screened calcein indicator. Titrate with EDTA standard solution (5.27) using the magnetic stirrer until the fluorescent green of the indicator completely changes to pink-purple with no residual fluorescence (see 5.2.7). Record the volume, V_1, of EDTA used in the titration to the nearest 0.02 mL.

NOTE. The EOTA solution end the use of potasium hydroxide, as described in 6.3.2.4 of BS 4661 : 1980 end 6.2 of BS 4550 : Part 2 : 1970, are acceptable alternatives when determining calcium oxide. Other indicators which give clear end-points, e.g. HSN, may be used.

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

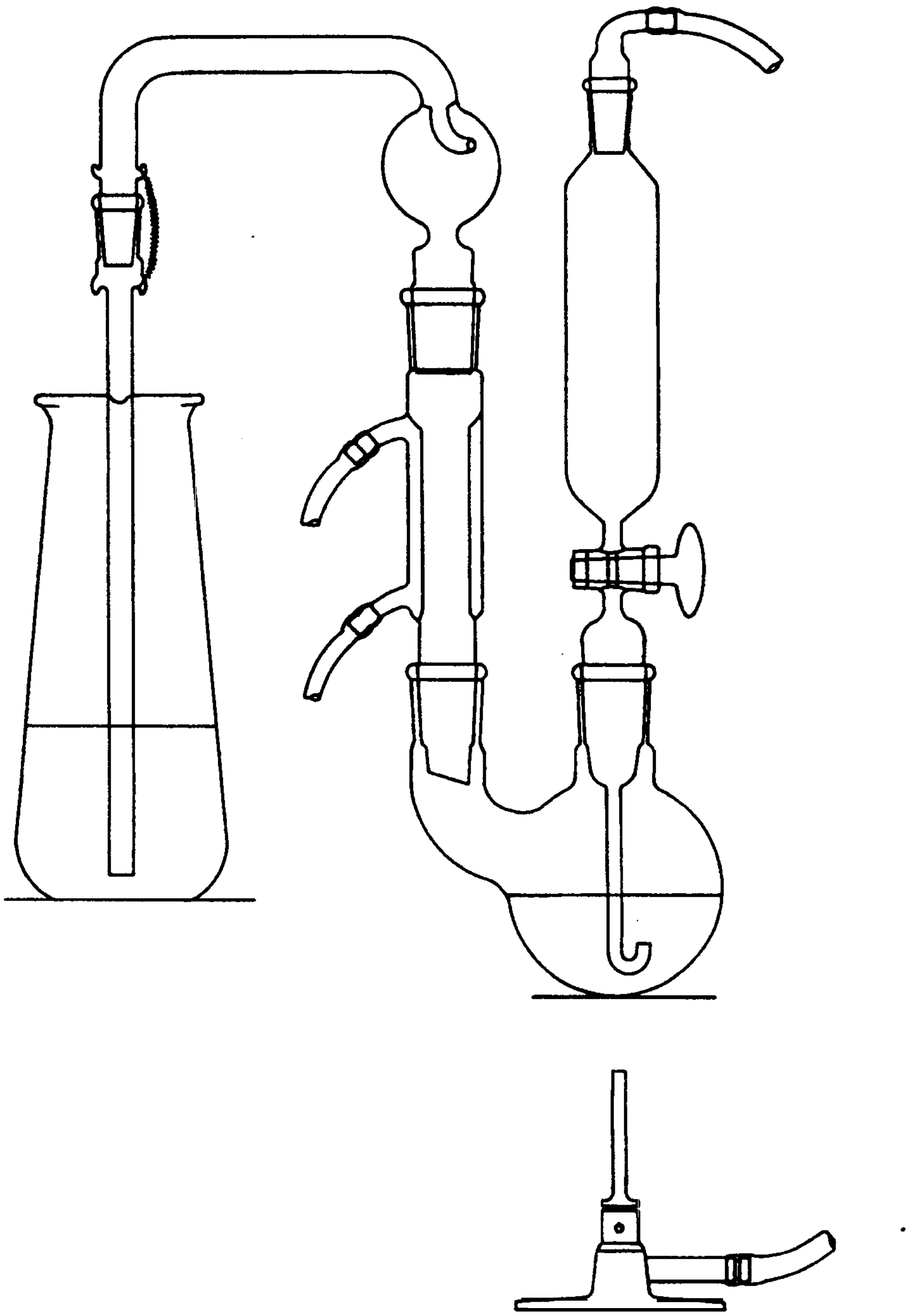
$$D = \frac{2V_1E}{Ma}$$

where

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

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

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



Figun 2. lypicai appratus for determination of content of sulphur as sulphide

5.5 Determination of insoluble residue, soluble silica and calcium oxide

5.51 *Extraction of soluble silica and preparation of stock test solution.* Weigh 5 ± 0.005 g of the analytical sample as prepared in 4.5.2 into a 250 mL polypropylene beaker and carefully add 100 mL of dilute hydrochloric acid (1+9). When a limestone aggregate is known to be present or when vigorous effervescence on adding the acid indicates its likely presence, add a further 10 mL of concentrated hydrochloric acid to the stirred suspension. Using a magnetic stirrer with a plastics covered magnetic follower, or other suitable mechanical stirrer, stir the suspension at room temperature for 20 min. Allow to settle and decant the liquid through either a medium ashless filter paper (see note to 5.3.1) or a paper pulp pad supported on a perforated cone or similar, with or without suction. Wash the residue in the beaker with three 25 mL portions of dilute hydrochloric acid (1+49) pouring the washings through the same filter paper. Retain this first filtrate.

NOTE 1. The rate of filtration is improved by adding 1 mL of polyacrylamide solution (5.2.10) 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.

Place the filter paper plus any residue it contains back into the beaker containing the residue and add 100 mL of the sodium carbonate solution (5.2.12). Place the beaker on a boiling water or steam bath (5.3.5) for 15 min. stirring occasionally with a plastics rod to break up the filter paper.

Transfer the contents of the beaker on to a medium hardened ashless filter paper (see note to 5.3.1) or a paper pulp pad supported on a perforated cone or similar, taking particular care to recover all the residue adhering to the sides of the beaker and the stirrer blade or magnet. Wash six times with the hot ammonium chloride solution (5.2.2), twice with hot dilute hydrochloric acid (1+49) and twice with hot water, the volume of each of the washings being about 25 mL. Allow each washing to drain before continuing with the next. Retain this second filtrate and the washings.

NOTE 2. To reduce tha possibility of silica being introduced into the solution the filtrate should be collected in a plastics beaker.

Add 10 mL of concentrated hydrochloric acid 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. Reserve the combined filtrates for treatment as described in 5.5.3.

5.52 *Determination of insoluble residue.* To determine the insoluble residue, 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 5.6). Allow the crucible and residue to cool to room temperature in the desiccator (5.3.6) and weigh them. Calculate the insoluble residue content of the analytical sample as a percentage to the nearest 0.1 % from the expression:

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

5.5.3 *Determination of soluble silica. Evaporate the* combined filtrates and washings on a boiling water or steam bath until not more than approximately 5 mL remain. Add 10 mL of dilute hydrochloric acid (1 + 1). half of an ashless filter tablet and 5 mL of the polyethylene oxide solution (5.2.11) stirring thoroughly during and after each addition. Stand for 5 min and filter through a medium ashless filter paper (see note to 5.3.1). Quantitatively transfer all the solids to the filter and wash thoroughly with hot water. 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 5.5.4.

NOTE 1. The stock test solution may also be used for other determinations as required, e.g. sulphate, magnesia, etc.

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 1200 ± 50 °C until constant mass is achieved (see 5.6). Allow the crucible and residue to cool to room temperature in the desiccator and weigh them. Calculate the soluble silica content of the analytical sample as a percentage to the nearest 0.1 % from the expression :

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

NOTE 2. The purity of the ignited silica precipitate can be ehackad by evaporating it in the crucible with 10 mL of hydrofluoric acid containing 5 drops of sulphuric 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 nsidue. When the two masses differ by more than 0.005 g it is recommended that the determination (5.5) 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.

5.5.4 *Determination of calcium oxide.* Pipette a 25 mL aliquot of the stock test solution from 5.5.3 into a 250 mL beaker or flask and dilute to about 150 mL. Add 10 mL of the dilute triethanolamine solution, 10 mL of the sodium hydroxide solution and about 0.15 g of screened calcein indicator. Titrate with the EDTA standard solution and calculate the calcium oxide content of the analytical sample as described in 5.4.

5.6 Determination of loss on ignition

Weigh 1 g to 2 g of the analytical sample as prepared in 4.5.2 into a crucible which has been previously ignited and weighed. Place the crucible in a furnace and, to avoid mechanical loss of material, cover the crucible with a lid and raise the temperature slowly to 925 ± 25 “C. 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. Allow the crucible to cool to room temperature in the desiccator and weigh. Repeat the ignition, cooling and weighing until constant mass is achieved as shown by successive weighings giving a difference in mass of less than 0.9005 g. Calculate the loss on ignition of the analytical sample as a percentage to the nearest 0.1 % from the expression :

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

5.7 Determination of carbon dioxide

Where it is considered useful to have knowledge of the carbonate content of the concrete, use the following method.

Assemble the apparatus (5.3.7) 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. Ensure that the tap funnel is acid-free. Accurately weigh approximately 02 g of the analytical sample as prepared in 4.5.2 into a duplicate reaction flask, replace the empty reaction flask by the one containing the sample and continue to pass gas free from carbon dioxide. Weigh the two absorption tubes and connect to the apparatus. Disconnect the gas inlet and place 30 mL of concentrated orthophosphoric acid (5.213) into the tap funnel.

Open the tap and reconnect the gas inlet; the gas pressure will force the acid into the reaction flask. After effervescence has ceased, slowly raise the temperature of the contents of the flask to boiling and boil for 5 min. Allow to cool for 15 min while maintaining the gas flow. Detach the absorption tubes, transfer to the balance case and weigh after 30 min.

To determine the ‘blank’ of the apparatus, which should be less than 1 mg, carry out the procedure as above 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 the whole procedure.

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

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

5.8 Determination of sulphur as sulphide

NOTE. This determination is only made when the slag content of a concrete containing a ground granulated blastfurnace slag of known composition as a reactive addition is required. When other constituents of the concrete contain sulphides, the method cannot be used. Occasionally, when analysing hardened concrete, not all the sulphide is recovered and so the slag content is underestimated. When this occurs, it is shown by a significant difference between the ordinary Portland cement 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 aggregte, or determined trom the aluminium oxide content and from the soluble silica content, for concretes containing significant quantities of calcium oxide in the aggregate.

Assemble the apparatus described in 53.8. Place 15 mL of the ammoniacal zinc sulphate solution (5.2.18) and 285 mL of water in the beaker. Weigh out 5.0 g of the analytical sample as prepared in 4.5.2 into the round bottom flask, add 2.5 g of stannous chloride (5.2.17) and disperse in 50 mL of water. Ensure that the glass outlet tube is immersed in the zinc sulphate solution. By means of the separating funnel add about 50 mL of dilute hydrochloric acid (1 + 1). and carefully close the tap of the separating funnel. Connect the inert gas supply, open the tap and adjust the flow to about 10 mL/min. Heat the contents of the flask to boiling and boil gently for 5 min or 6 min. Stop heating and wait 5 min or 6 min before shutting off the gas supply. Disconnect the delivery tube, which then serves as a stirrer. After cooling the contents of the beaker to about 20 °C, pipette into the beaker an excess of volume of the standard potassium iodate solution, 20 mL is sufficient when the sample contains up to about 0.15 % sulphide, and record the volume added, V_2, to the nearest 0.02 mL. Add 40 mL dilute hydrochloric acid (1+1), mix and immediately titrate with the sodium thiosulphate solution (5.2.16) until the colour of the liquid becomes a pale yellow. Then add 2 mL of the starch solution and continue the titration until the colour changes from blue to colourless. Record the volume of thiosulphate required, V_4, to the nearest 0.02 mL.

Calculate the sulphide content *S* of the analytical sample as a percentage to the nearest 0.01 % from the expression:

$$S = \frac{(V_2 - V_4 T)}{20 Mb}$$

where

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

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

T is the factor from the standarditation of the sodium thiosulphate solution;

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

5.9 Calculation of cement and aggregate content

5.9.1 General. Determine the calcium oxide content (see 5.4 or 55.4) and the soluble silica content (see 5.5.3) of the analytical sample as prepared in 4.5.2 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.

When the two cement contents thus obtained are within 1 % *(m/m)* of each other or, when converted to kg/m’, 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. 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 analysts' 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. may prove helpful in making the assessment. If such an assessment is not possible, indicate that the lower cement content is to be preferred.

Where it is known that the aggregate has a calcium oxide content of less than 0.5 %, the determination of cement content by way of the soluble silica content is not obligatory. but nevertheless recommended, and the wment content wn 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 are not recommended.

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 4.5.2, 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 5.9.2. If possible, the constituent chosen shall generally form a greater proportion of the cement than of the aggregate.

NOTE 2. When samples of the aggregate are not available, petrographic methods may be applicable, see ASTM CB56[1].

5.9.2 Using determined values for cement and aggregate. Assuming that the combined water of hydration of the concrete is 023 x 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 wment content or the aggregate/cement ratio from the following expressions:

(a) the percentage cement content C_1 to the nearest 0.1 %

$$C_1 = \frac{c - b}{a - 1.236} \times 100$$

(b) the cement content C_2 to the nearest 1 kg/m³

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

(c) the percentage aggregate content F to the nearest 0.1 %

$$F = \frac{a - 1.23c}{a - 1.236} \times 100$$

(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 wlcium oxide or soluble silica content of the aggregate (in %). see note 1;

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

NOTE 1. If the overall aggregate grading is not to be determined (see 6.4.31. 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 deign+. The weighted mean calcium oxide or soluble silice content of the aggregate should be calculated and these values used in the equations.

NOTE 2. If a ground granulated blastfurnace slag of known composition, e.g. d % calcium oxide, e % soluble silica and f % sulphide, has been incorporated in the concrete es a reactive addition, but the concrete does not contain blastfurnace slag aggregate end the sulphide content of the analytical sample has been determined (see 5.8), then assuming no sulphide in the ordinary Portland cement. the percentage slag content P is calculated from the expression:

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

where

S is the sulphide 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 ordinary Portland cement and aggregete and these corrected values are used to calculate the ordinary Portland cement content.

NOTE 3. 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 estimetion of the ordinary Portland cement content.

5.9.3 Using assumed values for cement and/or aggregate. Determine the calcium oxide (see 5.5.4) and soluble silica (see 5.5.3) contents of the analytical sample as prepared in 4.5.2. Make the best assumptions for the analysis of the Cement and/or aggregate and calculate the cement content using the expressions in 5.9.2.

NOTE. Reference to appendix B will indicate suitable values for calcium oxide and silice for typical cements. For Portland cements complying with BS 12 them will be little loss in accuracy in assuming that the calcium oxide content is 64.5 % but rather greater loss in accuracy in assuming that the silice content is 20.2 %. For some aggregates it may 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.

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

5.9.4 Alternative method for aggregate content based on insoluble residue values

NOTE 1. This test cennot be used if a composite cement, e.g. Portlend pozzolana cement having a significant insoluble residue, more than 10 % has been used.

Determine the insoluble residue (see 5.5.2) of the analytical sample as prepared in 4.5.2, and of the aggregate sample(s). Then assuming no insoluble residue in the cement, calcuate the aggregate content as a percentage from the expression:

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

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[1] Available from the Library, BSI, Linford Wood, Milton Keynes MK14 6LE.

NOTE 2. When samples of the aggregate are not available for analysis there may be little loss of accuracy in assuming that with essentially insoluble aggregate the percentage insoluble residue of the analytical sample (see 5.5.2) corresponds to the percentage aggregate content.

6 Aggregate grading

NOTE. This aggregate grading test is only applicable to concrete containing aggregates essentially insoluble in dilute hydrochloric acid.

6.1 Reagents

NOTE. See 5.2.1.

6.1 .1 *Ammonium chloride solution,* 1 g/L, see 6.2.2.

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

6.1.3 *Polyethylene oxide solution, 2.5* g/L, see 5.2.11.

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

6.2 Apparatus

6.2.1 *5.0 mm test sieve,* complying with BS 410, fitted with a lid and receiver.

6.2.2 75 µm test sieve, complying with BS 410.

NOTE. The sieves specified in BS 812 for sieving aggregate finer than 5.0 mm may also be required.

6.2.3 Chisel-edged hand tool.

NOTE. Small electrically operated hand tools may be of assistance.

6.2.4 Device for sample reduction, such as a riffle box.

6.2.5 1 L glass *beakers.*

6.2.6 Furnace, controlled to maintain a temperature of 400 ± 50 °C (see note 1 to 6.3).

6.2.7 Container, of nickel, stainless steel or refractory material of a size large enough to hold the sample and to fit in the furnace (see note 1 to 6.3).

6.3 Procedure

Use the sub-sample, portion B, obtained according to 4.6.1.

Separate the weighed sub-sample of concrete into coarse and fine fractions using the 5.0 mm sieve (6.2.1).

NOTE 1. Break-down of the concrete without fracture of the aggregate may be assisted by heating in a furnace (6.2.6) to 400 ±50 °C for 1 h to 16 h in a nickel, stainless steel or refractory container (6.2.7). If the matrix is still insufficiently weakened, thoroughly soak the concrete in water and re-heat. Microwave heating may also be appropriate.

Carefully clean the coarse aggregate portion of adhering matrix by chipping the individual pieces with the chisel-edged tool (6.2.3). Pass the material so removed through the 5.0 mm sieve. When all the material larger than 5.0 mm has been cleaned, treat it with dilute hydrochloric acid (1+9) (6.1.2) until the aggregate is free from adhering cement, rinse with water, dry, weigh and record the mass, m_1.

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 5.0 mm sieve during the mechanical cleaning of the coarse aggregate. Weigh and record the mass, m_2.

Using a riffle box (6.2.4), or by equivalent means, subdivide the weighed combined material passing 5.0 mm to produce a sub-sample of 50 g to 100 g, weigh and record its mass, m_3.

Transfer the weighed sub-sample, m_3, to a 1 L beaker (6.2.5), add 500 mL of dilute hydrochloric acid (1+9) and heat to about 50 °C. Maintain at this temperature, with occasional stirring, until disintegration appears to be complete. Add, whilst stirring, 5 mL of the polyethylene oxide solution (6.1.3). Allow to settle and decant the liquid through a pre-weighed fast hardened ashless filter paper (see note to 5.3.1). 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. 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 (6.1.4). Simmer gently for 15 min taking care to avoid loss by frothing. Transfer the contents through the 75 µm test sieve (6.2.2) into the original filter paper. Wash six times with the ammonium chloride solution (6.1.1). twice with dilute hydrochloric acid (1 + 9) and twice with hot water. Dry separately the sieve residue and the filter paper plus its contents, at 105 ± 5 °C, allow them to cool and weigh separately. 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 2. If required, a sieve analysis using the sieves specified in BS 812 : Part 103 may be carried out on the dried fine aggregate.

NOTE 3. When the coarse aggregate is visually 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 already described, before the weighings to obtain m_4 or carrying out the sieve analysis.

6.4 Calculations

6.4.1 Fine aggregate grading. Calculate the cumulative percentage passing each of the sieves used, including that with a 75 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 found, particularly with respect to the fraction passing 75 µm. cannot be used to assess compliance of the fine aggregate with BS 882.

6.4.2 Coarse aggregate grading. The separated coarse aggregate cannot be used to assess compliance of the grading of the coarse aggregate with BS 882.

6.4.3 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.

7 Original water content

7.1 Applicability

7.1.1 General. The test described in this clause will only give acceptable results if the concrete is sound and in no way damaged, either physically or chemically. The method is not suitable for poorly compacted concrete, for semi-dry compacted concrete, for air-entrained concrete or for aerated concrete. With some aggregates, often manufactured, which are exceptionally porous or contain appreciable amounts of combined water, the corrections to be applied are so great as to render the results of doubtful value.

NOTE 1. The application of the method to concretes containing hydraulic binders other then ordinary Portland cement requires special consideration.

NOTE 2. Unreliable results may be obtained when concrete older than 5 years is tested owing to changes in the pore structure of such concretes.

By the original water content is meant 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;

(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 water/cement ratio and this entails the determination of the cement content of the prepared sample.

NOTE 3. It should be noted that the prepared sample, while representative for the water/cement ratio of the whole sample, will not necessarily be representative for the cament content.

The water/cement ratio is expressed in two ways:

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

(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 will include contributions from the aggregates present and these shall be taken into account.

7.1.2 *When aggregate control samples are available.* Always make the correction for the combined water content of the aggregate (see 7.5.5).

Correcting for aggregate porosity (see 7.5.4) will give the original free water/cement ratio, while not making the correction will give the original total water/cement ratio, provided that the aggregate pores were filled with water when the concrete had set. When there is some doubt 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 doubt, report both free and total water/cement ratios.

7.1.3 *When aggregate control samples are not available.* Assume that the combined water of hydration of the concrete is

$$0.23 \times C_1$$

NOTE. The range of values found for the combined watar of hydration of Portland caments complying with BS 12, BS 146, BS 1370 or BS 4027 is between 0.20 end 0.25 of the cement, for full hydration. For vary young concrates (less than 28 days) special considerations may apply.

The capillary porosity of the aggregate shall be taken to be equivalent to the water absorption value of the aggregate when this is reliably known (see 7.6.2) 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.

7.2 Reagent

7.2.1 *1.1, 1-trichlorethane.*

7.3 Apparatus

73.1 *Vacuum desiccator,* 250 mm diameter.

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

7.3.3 *Apparatus for the determination of combined water content.* The apparatus consists of a small absorption vessel, containing dried magnesium parchlorate, 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 100 °C, bright red heat. The exit from the combustion tube shall be packed with silver wool to remove any hydrogen sulphide evolved. If silicone rubber connections are used it is advisable to protect them with heat reflectors. The gas leaving the combustion tuba 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 sulphuric acid is fitted after the exit end of the second absorption tube to allow the gas flow to be observed. A typical apparatus is shown in figure 3.

7.4 Determination of capillary porosity

Prepare the sample by cutting a slice as described in 4.3, dry it at 105 ± 5 °C for not less than 16 h and allow it to cool to room temperature in the desiccator (53.6).

Weigh the dried slice and immerse in 1.1,1-trichlorethane (7.2.1) contained in the vacuum desiccator (73.1).

Caution. Avoid breathing the vapour and contact of the liquid with the skin and eyes. Gloves shall be worn when handling the samples.

NOTE 1. Polyethylene gloves are recommended.

Reduce the pressure in the vessel to less than 13.5 kPa (100 mmHg) by means of the water pump or vacuum pump (7.3.2)[*].

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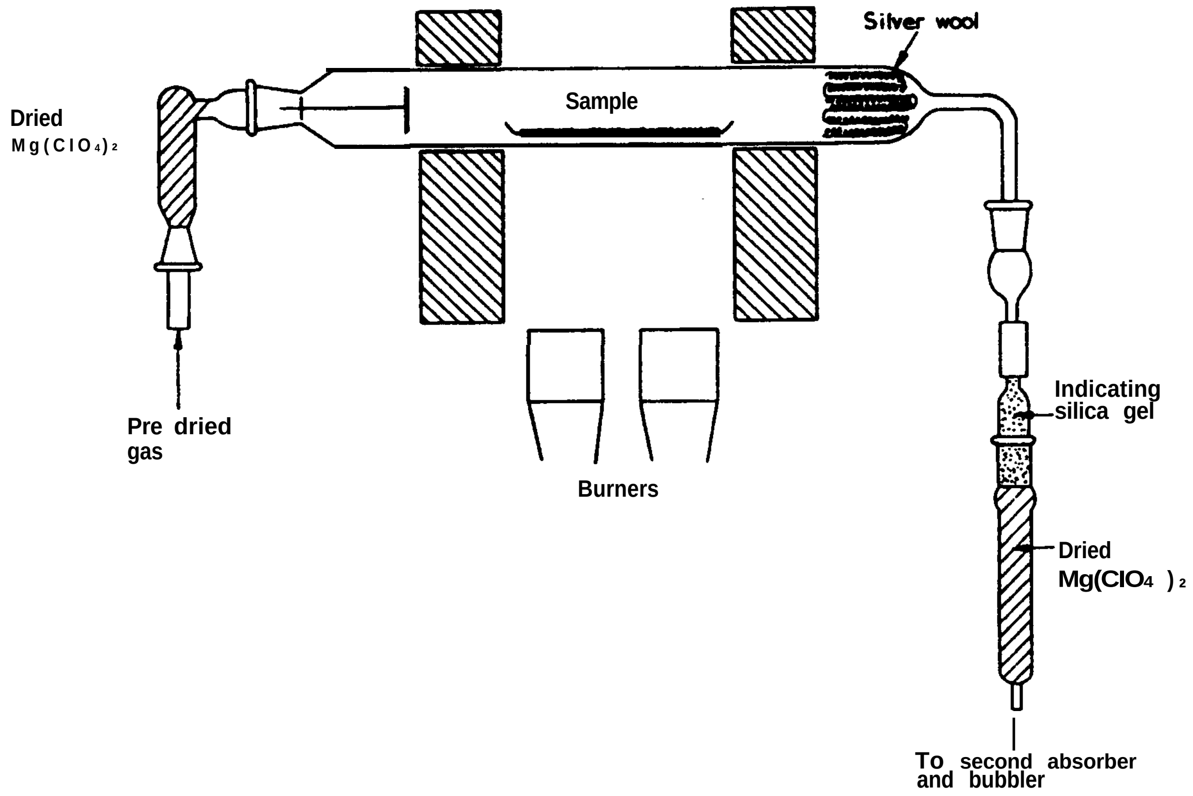
[*] It may be necessary to protect the vacuum pump against the effects of solwnt vapour by fitting a cold trap or similar device.



Figun 3. Typical apparatus for determination of water content

The air then 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 vassal is cautiously given a sharp tap.

NOTE 2. The time required to remove the air may be several hours. 'Boiling' of the 1 , 1,1 -trichlorethane induad by low pressure with continued evacuation should not be confused with air evolution and is normally recognisable by the tendency of large bubbles to appear a a few points on the concrete surface.

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

Remove the sample from the 1 , 1,1 -trichlorethane. Allow excess superficial liquid to drain away and quickly wipe excess solvent from the surfaces of the sample.

Without delay, place the saturated sample in a weighed polyethylene bag, seal to prevent loss of liquid by evaporation, and weigh. Calculate the mass of 1 ,1 ,1 -trichlorethane required to fill the pores of the concrete and from this mass derive the equivalent mass of water assuming that the relative density of the 1 ,1 ,1 -trichlorethane is 1.33.

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

$$Q = \frac{\text{mass of solvent absorbed} \times 100}{1.33 \times \text{mass of dried concrete sample}}$$

7.5 Determination of combind water and cement content

7.5.1 *Sample preparation.* Remove the slice from the polyethylene bag. 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. Treat the slice using the procedures described in 4.5.2 *to* produce a representative powdered sample of not less than 20 g all passing a 150 pm sieve and determine the combined water and cement contents of this powdered material by the procedures detailed in 7.5.2 to 76.5.

7.5.2 Determination of combined water content. Using the apparatus detailed in 7.3.3, determine the combined water directly by igniting the powdered sample at 1000 ± 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.

Purge the apparatus of residual moisture by passing dried gas through the combustion tube, heated to 1000 °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. 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.

When the combustion tube has cooled to below 100 °C, weigh the absorption tubes and fit into the train. Weigh accurately approximately 1 g of sample into a combustion boat previously ignited to constant mass at 1000 °C and promptly insert into the combustion tube from the air inlet end. Replace the connection and adjust the gas flow to about 3 bubbles per second at the bubbler. Heat, cautiously at first, so as to maintain the temperature at 1000 °C for 30 min. Remove the source of heat and continue to pass the gas for a further 15 min. Disconnect the absorption tubes, transfer to the balance case and weigh after 15 min.

To determine the 'blank' of the apparatus, which should be less than 1 mg, carry out the procedure from the beginning 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. Then repeat the blank determination. If a value greater than 1 mg is still obtained, replace the absorbents and repeat the whole procedure.

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

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

7.5.3 Determination of cement content. Determine the cement content of the prepared sample by the appropriate method as used for the analytical sample (see 5.9).

7.5.4 Determination of aggregate porosity. Weigh about 1 kg of the dried coarse aggregate and determine the capillary porosity by the procedure given in 7.4 but calculating it as a percentage *q*. For convenience, enclose the aggregate in a plastic mesh bag or support it on a sieve. Dry the surface of the aggregate before reweighing by shaking off the excess liquid and rapidly rubbing in a cloth.

7.5.5 Determination of aggregate combined water content. Heat the solvent-saturated aggregate to constant mass (as in 7.5.1) at 105 ± 5 °C and crush to pass a 150 pm sieve. Determine the combined water content by the procedure given in 7.5.2 but calculating it as a percentage Y.

7.5 Calculation of the original water/cement ratio

7.6.1 *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 the

original free water content = corrected capillary porosity + corrected combined water content or

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

$$W_f = Q + X \quad \frac{F}{100}(q + Y) \text{ (in \%)}$$

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

original total water content = uncorrected capillary porosity + corrected combined water content or

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

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

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

where

Q is the capillary porosity of the sample (in %) (see 7.4);

q is the capillary porosity of the aggregate (in %) (see 7.5.4);

X is the combined water content of the sample (in %) (see 7.5.2);

Y is the combined water content of the aggregate (in %) (see 7.55);

C_1 is the cement content of the concrete (in %) (see 7.5.3);

R is the aggregate content of the concrete (in %) (see 5.9.2).

7.6.2 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

$$\text{original free water/cement ratio} = \frac{Q}{C_1} - \frac{q'F}{100\,C_1} + 023$$

NOTE. If *q'* is not reliably known then only the original total water/cement ratio cm be reported.

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

8 Type of cement

8.1 General

Differentiation between the various types of cement that may have been used in a concrete presents problems of varying complexity. In favourable circumstances it may be possible to differentiate between various cements by analysing for an element or elements peculiar to the cement in question after careful separation of the cement matrix, for typical analyses of various types of cement see appendix B.

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

The test cannot be used to confirm that the cement used in the concrete did or did not comply with a cement specification. However, the test may help to determine compliance or otherwise with any requirement referring to general cement type in a concrete specification.

8.2 Separation and analysis of matrix

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 75 pm sieve. If insufficient sample is obtained, further very careful breakdown is necessary.

Analyse the very fine material so obtained for insoluble residue (see 5.5.2) and loss on ignition (see 5.6) and, for example, soluble silica (see 5.5.3). calcium oxide (see 5.5.4). alumina, ferrous oxide, magnesium oxide, sulphuric anhydride, etc. 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

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

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

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

8.3 Examination under a microscope

8.3.1 General

NOTE 1. This examination should be made by a microscopist with experience of cement and concrete materials.

The test is usually carried out to establish whether ordinary Portland cement or sulphate-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 gains greater than 20 µm in size.

NOTE 2. In older concretes which have been kept wet, e.g. foundations, it may prove impossible to find sufficient anhydrous grains.

NOTE 3. It will frequently be possible to determine whether either blastfurnace slag or pulverized-fuel ash is present. Howewr, these identifications sometimes require the additional examination of thin sections in transmittad light and such a procadum is outside the scope of this Part of BS 1881.

8.32 Reagents

8.3.2.1 Mounting resin. An epoxy or polyester resin which sets rigidly when mixed with an appropriate hardener.

8.3.2.2 Silicon carbide powder, medium grade, e.g. 26 pm.

8.3.2.3 Non-aqueous liquid, e.g. paraffin.

8.3.2.4 Diamond paste, of grades 14 pm, 6 pm, 3 pm, 1 pm and 0.25 pm.

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

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

8.3.2.7 Methylated spirits or ethanol.

8.3.2.8 *Hydro fluoric acid.*

8.3.3 *Apparatus*

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

8.332 *Microscope.* A reflected-light microscope using magnifications up to X BOO.

8.3.3.3 Cylindrical mould, at least 25 mm in diameter, e.g. a plastics tube.

8.3.4 Preparation of the sample. Use the sample, portion B, obtained according to 4.5.1.

Break up the concrete sample and select at least 10 nominal 5 mm sized pieces of cement-rich-matrix as the test portion. Dry the test portion at a temperature not exceeding 105 °C, allow to cool and then embed the pieces in a low viscosity epoxy or polyester resin (8.3.2.1) by casting under vacuum into a cylindrical mould (83.33) and curing at the temperature recommended by the resin manufacturer.

NOTE 1. A cylindrical cast specimen of approximately 25 mm diameter is usually found to be suitable but larger or multiple specimens are also acceptable.

Section the cylindrical casting with a diamond saw lubricated with a non-aqueous liquid so that cut surfaces of the pieces are exposed on the sawn surface, and wash immediately using a non-aqueous solvent, e.g. acetone* (83.25). Grind the sawn surface with silicon carbide powder (8.3.2.2) lubricated with a non-aqueous liquid such as paraffin, or other suitable oil (8.3.2.3), to remove saw marks. Remove all traces of grinding powder.

NOTE 2. Treatment with paraffin or industrial methylated spirits in an ultrasonic bath is effective.

Carefully polish with progressively finer diamond compounds (8.314) in an oil soluble base on a rotary lap-plate (8.3.3.1) with a suitable surface. After a final polish with 025 pm diamond compound, clean the surface using a non-aqueous cleaning agent and dry with tissue. The grinding and polishing routine shall be adjusted, particularly in terms of speed and duration of lapping, to minimize the plucking of cement particles out of the polished surface.

NOTE 3. It is recommended that 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.

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*Prolonged immersion in acetone can soften epoxy and polyester resins.

8.3.5 Cement grain identification. Examine the polished surface through a reflected-light microscope using magnifications up to ×800. If insufficient grains of anhydrous cement are found (see 8.3.1). either prepare further polished samples as in 8.3.4 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 may 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.

Chemical etching of the grains reveals mineral phases characteristic of the type of cement present. It is permissible to use two alternative etchants.

(a) Immerse the polished surface in the potassium hydroxide solution (8.3.2.6) at 30 °C for 10 s to 20 s, then wash immediately in industrial methylated spirits or ethanol (8.3.2.7). The etching shall be controlled to give a bluish-grey colour to the tricalcium aluminate; the silicates remain grey, and the ferrite appears white.

(b) Expose the polished surface to hydrofluoric acid vapour (8.3.2.8) for 2 s to 5 s until the hexagonal alite is coloured straw-brown; the rounded belite will appear 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 3. It is sometimes difficult to distinguish these two compounds particularly at low magnification, i.e. less than X 500.

Caution. Hydrofluoric acid is extremely dangerous and great care is required in its use, even in small quantities. Hydrofluoric acid vapour etching shall be performed in a fume cupboard. The vapour can damage microscope lenses and excess vapour shall be permitted 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.

Using the compositional information given in appendix C, and making reference to the control specimens if prepared, identify the apparent type of cement represented by each anhydrous grain examined. The cement type present in the concrete sample will be that represented by at least 80 % of the anhydrous grains examined.

If the dominant cement type represents fewer than 80 % of the grains examined, repeat the whole procedure 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 4. 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.

9 Type of aggregate

9.1 General

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.

NOTE. Guidance notes for the petrographic examination of hardened concrete have been published as ASTM C856*.

9.2 Procedure

Prepare a broken, or preferably a sawn, surface of the sample (see 4.5.3). Identify the general character of the aggregate and compare the aggregate exposed with known samples whenever possible.

NOTE 1. Most carbonate aggregates may 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, them may nevertheless be some carbonate particles, or particles containing carbonate which will react with dilute acid, e.g. shell debris. Some dolomites do not react rapidly with cold dilute hydrochloric acid but will effervesce with warm dilute hydrochloric acid.

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

10 Other constituents

10.1 General

The analytical sample as prepared in 4.5.2 may be used for the determinations of chloride, sulphate and alkali contents. However, the test for alkali content may require a sample of separated fines to avoid aggregate interference (see 10.4). Since the chloride and sulphate contents are usually reported in relation to the cement content of the concrete, such a sample, in which the cement matrix has been concentrated, may present some advantages in all these tests.

A suitable procedure is to break the concrete sample and to remove fine material by sieving through a 150 µm sieve. Further breaking and sieving is carried out until a sufficient quantity of fines has been produced. The less the aggregate is damaged the higher the cement content of the fines. The cement content of this separated sample has to be determined using the procedure described in 5.9.

10.2 Determination of chloride content

NOTE 1. Alternative methods based on potentiometric titration are acceptable.

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*Available from the Library, BSI. Linford Wood, Milton Keynes MK14 6LE.

NOTE 2. Salts of thiocyanates and thiosulphates are increasingly used as accelerating admixturas in place of chlorides. These compounds can interfere with the determination of chloride content. Henca, a short period of boiling after adding nitric acid to the sample has been introduced into tha procedure in 10.22 to decompose these compounds. Quantitative methods for thiocyanates and thiosuphates are being developed.

10.2.1 *Reagents*

NOTE. See 5.2.1.

10.2.1.1 *Nitric acid,* relative density 1.4.

10.2.1.2 *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.

10.2.1.3 *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 I I I indicator, once a week or each time a determination of chloride content is made, if less frequent.

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

10.2.1.5 *3,5,5-trimethylhexanol (nonyl alcohol).*

10.2.2 Procedure. Weigh into a stoppered 500 mL conical flask 5 ± 0.005 g of the analytical (see 4.52) or separated (see 10.1) sample. Disperse with 50 mL of water and add 10 mL of nitric acid (10.2.1.1). Add 50 mL of hot water, boil for 4 min to 5 min and keep warm for 10 min to 15 min. (If the supernatant liquid is turbid, filter through a fast hardened ashless filter paper (see note to 5.3.1) and wash with hot water.) Cool to room temperature and add a measured excess of the silver nitrate standard solution (10.2.1.2).

Add 2 mL to 3 mL of 3.5.5-trimethylhexanol (10.2.1.5), stopper the flask and shake vigorously to coagulate the precipitate. Add 1 mL of iron I I I indicator solution (10.2.1.4) and titrate with the thiocyanate solution (102.13) to the first permanent red colour.

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

$$J = \left\{ V_5 - \frac{V_6 m}{0.1} \right\} \frac{0.3545}{M_c} \times \frac{100}{C_1}$$

where

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

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

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

m is the molarlty 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 ba used and the equation modifiad accordingly.

10.3 Determination of sulphate content

10.3.1 *Reagents*

10.3.1.1 *Hydrochloric acid,* relative density 1.18.

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

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

10.3.1.4 *Barium chloride solution,* 100 g/L. Dissolve 100 g of barium chloride in 1 L water.

10.3.1.5 *Methyl red indicator solution.* Dissolve 1 g of methyl red in 600 mL of methylated spirits and add 400 mL of water.

10.3.2 *Procedure.* Weigh into a 400 mL beaker 5 ± 0.005 g of the analytical (see 4.52) or separated (see 10.1) sample. Disperse with 50 mL of water and add 10 mL of concentrated hydrochloric acid (10.3.1.1). 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. Filter through a medium ashless filter paper (see note to 5.3.1). washing the residue thoroughly with hot dilute hydrochloric acid (1+49) (10.3.1.2). Add three drops of the methyl red indicator (10.3.1.5) and heat the filtrate to boiling. Just neutralize to yellow with the dilute ammonium hydroxide solution (10.3.1.3). Immediately add 1 mL of concentrated hydrochloric acid and then add dropwise to the boiling solution 10 mL of the barium chloride solution (10.3.1.4). If excess ammonium hydroxide was added, 1 mL of concentrated hydrochloric acid may not be sufficient to obtain the required acid solution and the barium sulphate precipitate will then be contaminated. In this case the test shall be repeated. 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. Filter through a slow ashless filter paper (see note to 5.3.1) and wash free from chlorides with hot water. 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 5.6).

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

$$G = \frac{L}{M_d} \times 34.3 \times \frac{100}{C_1}$$

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 sulphate (in g).

NOTE. The stock test solution (see 5.5.3) may be used for this datermination. Usa a 200 mL aliquot and proceed as before from the haating stage to boiling and neutralizing with dilute ammonium hydroxide solution. In the calculation, M_d will be 2 g.

10.4 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 below 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 10.1) and care taken to minimize contamination of the fines with aggregate.

10.4.1 *Apparatus.* Use the flame photometer described in 16.2.1 of BS 4550 : Part 2 : 1970.

10.4.2 *Reagents.* Use the reagents given in 1622 of BS 4550 : Part 2 : 1970.

10.4.3 *Calibration.* Calibrate the flame photometer by the method given in 16.2.3 of BS 4550 : Part 2 : 1970.

10.4.4 *Procedure.* Weigh into a 400 mL beaker, made from a material which will not yield sodium or potassium to the solution, 5 ± 0.005 g of the analytical sample (see 4.52) or 2 ± 0.005 g of the separated sample (see 10.1). disperse with 150 mL of water and add 20 mL of concentrated nitric acid. Boil the solution gently for 10 min. cool and quantitatively transfer to a 500 mL volumetric flask. Dilute to the mark with water, mix thoroughly and filter off about 190 mL through a dry medium ashless filter paper (see note to 5.3.1).

Pipette 25 mL of the filtrate into a 100 mL volumetric flask, add 10 mL of aluminium solution, dilute to the mark with cold water and mix thoroughly.

With the sodium light filter in place set the flame photometer to read 0 with scale-zero solution and 100 with scale-109 solution. Spray the sample solution and take several readings, checking, and adjusting if necessary, the 0 and 100 settings between each reading. Replace the sodium light filter by the potassium light filter and again set the flame photometer to read 0 with scale-zero solution and 100 with scale-100 solution. Spray the sample solution and take several readings, checking, and adjusting if necessary, the 0 and 100 settings between each reading.

If the sample gives a reading of more than 100 for either sodium or potassium, pipette 25 mL of the filtrate into a 200 mL graduated flask, add 20 mL of aluminium solution, dilute to the mark with cold water, mix thoroughly and respray.

10.4.5 *Calculation.* From the respective calibration graphs for sodium oxide and potassium oxide (see 10.4.3) 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 mg/L);

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 of more than 100 for either sodium or potassium and in consequence the filtrate was pipetted into a 200 mL graduated flask

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

Then the sodium oxide equivalent N_e as a % *(m/m)* of cement

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

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 may be higher then the true alkali content of the cement used due to contributions from sources other than the cement, particularly when the analytical sample (see 4.5.2) has been used for analysis.

The sodium oxide equivalent content of the concrete N_b is calculated in kg/m³ from:

(a) where the analytical sample (see 46.2) was used

$$N_b = \frac{(u + w \times 0.5581}{100} \times \rho_9$$

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/m³);

or (b) where a sample of separate fines (see 10.1) 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 5.9 (in %).

11 Report

11.1 General

The report shall affirm that the analysis was made in accordance with this Part of BS 1881. When alternative methods are used, e.g. see 5.1, the report shall include details of them and evidence that they are of equivalent accuracy. A copy of the certificate of sampling shall be provided with the report.

11.2 Information to be included in the report

11.2.1 *Obligatory information. The* following information shall be included in the report:

(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.;

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

11.2.2 *Additional information. Where* appropriate, the following supplementary information shall also be included in the report:

(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 done at the analyst's discretion;

(d) conclusions regarding the concrete quality where required.

Appendices

Appendix A Precision

Estimates of the repeatability and reproducibility of hardened concrete analysis for cement content are given in table 1. The data have been obtained for concretes made with ordinary Portland cement and may 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 5497 : Part 1. 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 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 5497.

The difference between two single results found on identical samples of the same concrete by one analyst within a short time interval will exceed 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 will exceed 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 averages of duplicate analyses of one analytical sample.

Table 1. Precision estimates

Type of aggregate	Range of cement contents investigated	oxide used to calculate cement content	*r*	*R*	σ_r	σ_L
	kg/m^3		kg/m^3	kg/m^3	kg/m^3	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

Appendix 6. Typical analyses of types of cement currently available in the UK

The following table 2 gives typical analyses of types of cement currently available in the UK.

Table 2. Typical analyses of types of cement currently available in the UK

	Ordinary and rapid-hardening BS 12	Sulphate-resisting BS 4027	Portland-blastfurnace BS 146 (see note 2)	Portland-pfa BS 6588 (see note 3)
	%	%	%	%
CaO	54.5	64.6	54.2	47.4
SiO_3	20.2	20.5	25.4	28.7 (15.5)
MgO	1.6	1.4	4.7	2.1
Al_2O_3	5.4	3.8	8.7	10.7
Fe_2O_3	2.6	5.0	1.2	4.9
SO_3	2.8	2.2	1.6	2.5

NOTE 1. These values are subject to a variation of at least ± 10 %, with the exception of CaO content in ordinary Portland cement or sulphate-resisting Portland cement which are subject lo a variation of approximately ± 3 %.

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

NOTE 3. Variations in the clinker and pulverizad-fuel ash (pfa) used and in their relative proportions may 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. Tha conditions of extraction may not bring all the other oxides into solution from the pozzolana although it may be expected that all the calcium will dissolve.

Appendix C. Mineral phase compositions of different types of cement

Portland cements not having a specification limit for tricalcium aluminate content, e.g. ordinary and rapid-hardening Portland cements complying with BS 12, generally have a volume ratio of ferrite to tricalcium aluminate of less than 2:1. For sulphateresisting Portland cement complying with BS 4027, the volume ratio of ferrite to tricalcium aluminate is generally more than 5:1. However, this ratio may not apply to sulphate-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 25 µm in size, may display an apparently anomalous phase composition. For this reason a concrete made with sulphate-resisting Portland cement complying with BS 4027, for example, may contain a significant number of grains showing larger than expected amounts of trialcium aluminate and thus having the appearance of ordinary Ponland cement. Finding a few apparently anomalous grains in the examination is therefore nut necessarily an indication of adulteration of the cement used.

Publications referred to

BS 12	Specification for ordinary end rapid-hardening Portland cement
BS 146	Specificetion for Portland-blastfurnace cement
BS 410	Specification for test sieves
BS 812	Testing aggregates
	Part 103 Methods for determination of particle size distribution
BS 882	Specificetion for aggregates from natural sources for concrete
BS 1370	Specifiition for low heet Portland cement
BS 1881	Testing concrete
	Part 101 Method of sampling fresh concrete on site
	Part 114 Methods for determination of density of hardened concrete
BS 4027	Specification for sulphate-resisting Portland cement
BS 4550	Methods of testing cement
	Part 2 chemical tests
BS 4551	Methods of testing mortars, screeds and plasters
BS5328	Methods for specifying concrete, including ready-mixed concrete
BS 5497	Precision of test methods
	Part 1 Guide for the determination of repeatability and reproducibility for a standard test method by inter-laboratory tests
BS 6100	Glossary of building and civil engineering terms
	Part 8 Concrete and plaster
BS8588	Specification for Portland pulverizd-fuel ash cement
ASTM C856	Standerd practice for petrographic examination of hardened concrete

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