

Testing concrete —

Part 124: Methods for analysis of hardened concrete

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- Association of Metropolitan Authorities
- British Aggregate Construction Materials Industries
- British Civil Engineering Test Equipment Manufacturers' Association
- British Precast Concrete Federation
- British Ready Mixed Concrete Association
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- Concrete Society
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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 It supersedes BS 1881-6:1971, which is withdrawn.

The publication as BS 1881-6 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 1980 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 sulphaaluminate 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 1881 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.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 24, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 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 the inside back cover.

2 Definitions

For the purposes of this Part of BS 1881, the definitions in BS 5328 and BS 6100-6 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 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 3.3).

3.2 Selection of sample

Observe the following general requirements in selecting the concrete sample.

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

- 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.

- 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 m³ 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 composition 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:

- the date and time of sampling;
- a description of the structure or component under investigation;
- the location of the positions from which the concrete samples were taken;
- the method of sampling;
- details of the concrete, in particular its age, where known;
- 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 µm will also be required if mechanical crushing and grinding is not used (see **4.5.2**).

4.1.4 *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 1881-114.

4.3 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 2 500 mm², 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 O: other aggregates.

4.5 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 three-quarters 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 sub-sample of 500 g to 1 000 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 5.7), 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 clear. Prepare the reagents by the following methods.

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

5.2.2 Ammonium chloride solution, 1 g/L.

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

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

5.2.4 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 200 °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 CaCO₃/1.7848.

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. Standardize 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 (5.2.3) 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).

5.2.10 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 note 1 to 5.5.1).

5.2.11 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.

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 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 sulphide. This potassium iodate solution is stable for several months.

5.2.16 Sodium thiosulphate solution, used when sulphur as sulphide is to be determined. Dissolve 7.4 g of sodium thiosulphate (Na₂S₂O₃·5H₂O) 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 solution from the expression:

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