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METHOD OF
SAMPLING, ANALYSIS AND TESTING OF CONCRETE
PART 3 - ANALYSIS OF
HARDENED CONCRETE

BUREAU OF CEYLON STANDARDS

METHODS OF SAMPLING, ANALYSIS AND TESTING OF CONCRETE

PART 3 : ANALYSIS OF HARDENED CONCRETE

SLS 262:Part 3:1975

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Sri Lanka.

Sri Lanka Standards are subject to periodical revision in order to accommodate the progress made by industry. Suggestions for improvement will be recorded and brought to the notice of the Committees to which the revisions are entrusted.

This Standard does not purport to include all the necessary provisions of a contract.

SRI LANKA STANDARD
METHODS OF SAMPLING, ANALYSIS AND TESTING OF CONCRETE
PART 3 : ANALYSIS OF HARDENED CONCRETE

FOREWORD

This Sri Lanka Standard has been prepared by the Drafting Committee on Methods of Sampling, Analysis and Testing of Concrete. It was approved by the Civil Engineering Divisional Committee of the Bureau of Ceylon Standards and was authorized for adoption and publication by the Council of the Bureau on 1975-04-02.

This standard has been prepared in 3 parts, issued separately as follows:

Part 1 - Methods of sampling fresh concrete and making test specimens

Part 2 - Methods of testing concrete

Part 3 - Analysis of hardened concrete.

This standard does not apply to concrete whose nominal maximum aggregate size exceeds 40 mm (1 1/2 in).

This part of the standard is limited to materials in which the binding agent is a hydraulic calcareous cement and the aggregates are natural rocks or inorganic artificial products.

The method specified in this standard gives the cement content, the aggregate proportions and the grading of the coarse aggregates.

All units given in this standard are SI units, with the appropriate inch equivalents in brackets.

The American, British and Indian Standards have been consulted in the preparation of this Standard Specification and acknowledgement is made of the assistance gained therefrom.

1 SCOPE

This part of the standard specifies the tests to be used on a sample of hardened concrete to provide some or all of the following information:

a) Test No. 1 - Cement aggregate content;

b) Test No. 2 - Original water content;

c) Test No. 3 - Bulk density;

d) Test No. 4 - Type of cement;

e) Test No. 5 - Type of aggregate; and

f) Miscellaneous - Chloride content, sulphate content and sulphoaluminate content.

2 SAMPLING AND TREATMENT OF SAMPLES

2.1 It is usual for the tests described in this standard to be applied only when there is some doubt about the quality of the concrete. The method of taking a representative sample depends on the type of the structure in question, which would vary widely. Hence, it is not possible to describe a general method which will be applicable in all cases.

It is strongly advised that, if there is dispute, no sample be taken for testing without agreement between the parties about the method of taking the sample and the quantity of material that is considered to be represented by the sample.

2.2 Sampling

The following general requirements should be considered:

a) The minimum linear dimension of the sample should be at least 5 times those of the largest aggregate particles;

b) The sample should be in a single piece;

c) All foreign matter and reinforcement should be avoided unless they are the subject of test.

Clearly label the sample with full particulars, including the date, exact place and method of sampling and any other relevant details, and enclose it in a polythene bag and seal or tie securely. Firmly fix a duplicate label to the outside of the bag.

Give all available information about the sample together with exact particulars of the information required. A suitable standard form is given in Appendix A.

2.3 Treatment of samples

2.3.1 If the original water content is to be determined, cut a slice from the sample with two parallel cuts, preferably with a diamond saw, avoiding as far as possible loss of material out of the smooth faces. The slice should be approximately 20 mm thick and have a single face area of not less than 10,000 mm^2. Place in an airtight container to prevent carbonation.

2.3.2 Reserve about one-third of the sample for further reference, and for previous determination of bulk density if required, and then break up the remainder of the sample into lumps not larger than about 50 mm size, avoiding as far as possible fracture of the aggregate. Dry in an oven at 105 $^{\circ}$C for 15 h to 24 h; if however, an estimate of sulpho-aluminate is required, dry the sample at 55 ± 5 $^{\circ}$C for 30 h to 48 h.

2.3.3 Cool and divide the dried sample into two approximately equal parts *A* and *B*.

2.3.4 Treat portion *A* to provide a representative subsample of not less than 20 g of ground material all passing a 150-µm CS fine mesh test sieve.

A suitable procedure is as follows:

Crush portion *A* until it all passes a 4.75-mm CS fine mesh test sieve and then divide into two equal parts. Crush the whole of one part to pass a 2.36-mm CS test sieve, and obtain a representative 1/8 part, for example, by quartering, and grind the whole of this to pass a 600-µm CS fine mesh test sieve. Again reduce to 1/8 and further grind to provide the sample all passing a 150-µm CS fine mesh test sieve.

2.3.5 Retain portion *B* in an airtight container for use in tests 1, 2, 4 and 5 and miscellaneous tests.

3 CEMENT CONTENT

Test No. 1 - Determination of cement content

3.1 About 7 kg of the sample of concrete is taken and the coarse aggregate is separated carefully using a hammer, chisel and a wire brush. Break down of the concrete without fracture of the aggregate may be assisted by heating to 550 ± 10 °C for 1 h to 16 h in a Nickel, stainless steel or refractory container. If the matrix is still insufficiently weakened, thoroughly soak the concrete in water and re-heat. The coarse aggregate is weighed and the proportion of coarse aggregate to cement mortar is determined. Let the mass of coarse aggregate be m_1 kg and mass of mortar m_2 kg.

3.2 The sample is sieved through 850-µm CS sieve to eliminate any fraction of coarse aggregate which may have been chipped out. What is retained on this sieve may be added to m_1. It is then reduced to about 100 g by quartering. The sample is then powdered using a porcelain pestle and mortar so as to pass through 300-µm CS seive. The sample is dried in the oven at 105 °C to 110 °C for two hours. About 10 g of this is ignited in the furnace at 800 °C for half an hour.

3.3 A quantity of approximately two grams of the ignited sample is weighed to an accuracy of one tenth of a milligrame in a watchglass. It is transferred to a beaker and dissolved in 250 ml of 1:10 hydrochloric acid. It is heated and filtered through No. 42 Whatmans filter paper or equivalent. The residue is washed two or three times with hot distilled water. It is then digested in 75 ml of one per cent sodium hydroxide solution. It is filtered through the same filter paper and washed with hot distilled water till free from chlorides.

3.4 Soluble silicon dioxide separation

3.4.1 The filtrate is evaporated to dryness on a water-bath and baked in the oven at 110 °C to 120 °C for one hour. 10 ml of concentrated hydrochloric acid is added to it and again evaporated and baked as before. This process is repeated again and finally 10 ml of concentrated hydrochloric acid and 10 ml of distilled water is added to it, warmed and filtered through No. 42 Whatmans filter paper or equivalent. The residue is washed with hot distlled water until washings are free from chlorides. The filter paper containing the residue is then ignited in a platinum crucible at 1000 °C for 30 minutes and weighed. Let the mass of the ignited residue silicon dioxide be m_3 g.

3.4.2 For more accurate determination of silicon dioxide, the silicon dioxide as obtained in the platinum crucible is moistened with a drop of distilled water and then with 2 drops to 3 drops of 1:1 sulphuric acid. Sufficient hydrofluoric acid (40 per cent) is added to just cover the silicon dioxide. It is evaporated to dryness under I.R. lamp or hot plate in the fume cupboard. It is then ignited in the furnace at 1050 °C for 30 minutes and weighed. The loss in mass is the mass of silicon dioxide (say a g).

3.5 Precipitation of mixed oxides (ferric oxide and aluminium oxide)

The filtrate obtained after separating silicon dioxide is evaporated to about 250 ml. A few drops of concentrated nitric acid is added and then concentrated ammonium hydroxide is slowly added to the boiling solution till the precipitation is formed. A few drops of ammonium hydroxide is added in excess and boiled for 1 minute to 3 minutes. The solution is filtered through No. 40 Whatmans filter paper or equivalent and the precipitate is washed with 1:10 ammonia solution several times.

3.6 Precipitation of calcium as calcium oxalate

The filtrate from 3.5 is evaporated to about 400 ml in a beaker and 50 ml of saturated ammonium oxalate solution is added to the boiling solution. The solution is boiled for 5 minutes to 10 minutes and allowed to settle down. It is filtered through a No. 40 Whatmans filter paper or equivalent and washed with one per cent ammonium oxalate solution and finally finally with hot distilled water. The precipitate in the filter paper is ignited at 1050 °C for half an hour and weighed. This gives the mass of calcium oxide (m_4 g).

3.7 Calculation of mixed proportions

All calculations are made on the ignited sample, an allowance of two per cent is given for loss of cement on ignition, and no allowance is made for the loss of fine aggregates (sand) on ignition.

When a sample of cement used in the work is not available the follwing assumptions are made:

a) Soluble CaO content in cement is 64.5 per cent.

b) Soluble SiO_2 content in cement is 21.4 per cent.

NOTE - If a sample of cement used is available, soluble calcium oxide and silicon dioxide should be determined separately.

3.8 When the fine aggregate is insoluble in dilute hydrochloric acid, the cement content should be calculated on the calcium oxide basis and silicon dioxide basis and the lower value is taken as shown in page 10.

3.8.1 *Calculation by calcium oxide basis*

Mass of CaO (in 2 g of mortar) $= m_4$ g

Mass of ignited cement $= \dfrac{m_4 \times 100}{64.5}$ g

Mass of non ignited cement $= \dfrac{m_4 \times 100}{64.5} \times \dfrac{100}{98}$ g

Proportions by mass of cement and sand

$$\frac{m_4 \times 100}{64.5} \times \frac{100}{98} : 2 - \frac{m_4 \times 100}{64.5}$$

Let this ratio be represented as C : S

3.8.2 *Calculation by silicon dioxide basis*

Mass of SiO_2 $= m_3$ g

Mass of ignited cement $= \dfrac{m_3}{21.4} \times 100$ g

Mass of non ignited cement $= \dfrac{m_3 \times 100}{21.4} \times \dfrac{100}{98}$ g

Proportion by mass of cement and sand

$$\left[\frac{m_3 \times 100}{21.4} \times \frac{100}{98}\right] : \left[2 - \frac{m_3 \times 100}{21.4}\right]$$

Let this ratio be represented as C : S

3.8.3 *Calculation of proportions by mass of cement:sand:coarse aggregate*

Mass of coarse aggregate in concrete $= m_1$ kg

Mass of cement mortar in concrete $= (7 - m_1)$ kg

Mix proportions of cement : sand : C : S

$$\text{Mass of cement in mortar (concrete)} = \frac{(7 - m_1) \times C}{C + S} \text{ kg}$$

$$\text{Mass of sand in mortar (concrete)} = \frac{(7 - m_1) \times S}{C + S} \text{ kg}$$

Cement : Sand : Coarse aggregate

$$\frac{(7 - m_1) \times C}{(C + S)} : \frac{(7 - m_1) \times S}{(C + S)} : m_1$$

3.9 When the fine aggregate is soluble in dilute hydrochloric acid, (for example, calcareous such as lime, limistone or dolomitic sand), the cement content is calculated on the silicon dioxide basis only.

3.9.1 If some lime is suspected in the mortar, the cement content is calculated on the silicon dioxide basis. Using the total calcium oxide value the amount of lime added can be determined.

$$\text{Mass of cement calculated from } SiO_2 \text{ basis} = \frac{m_3 \times 100}{21.4} \times \frac{100}{98} \text{ g}$$

$$\text{Mass of soluble CaO from cement alone} = \frac{m_3 \times 100}{21.4} \times \frac{100}{98} \times \frac{64.5}{100} \text{ g}$$

Total mass of CaO obtained from the mortar sample $= m_4$ g
(Value obtained from CaO basis is taken)

$$\therefore \text{ Lime added } = m_4 - \left[\frac{m_3 \times 100}{21.4} \times \frac{100}{98} \times \frac{64.5}{100}\right]$$

3.10 When the fine aggregate is partly soluble in dilute hydrochloric acid

The above method is not reliable unless separate determination of soluble calcium oxide and silicon dioxide contents are carried out with the fine aggregate and due allowances are made for them, that is : soluble calcium oxide and silicon dioxide contents must be substracted from the total calcium oxide and silicon dioxide obtained with a sample of mortar. The method becomes meaningful only if a free sample of fine aggregate is available along with the sample of concrete.

4 ORIGINAL WATER CONTENT

Test No. 2 - Test for original water content

The test described below 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 nor for semi-dry compacted concrete. With aggregates (often artificial) 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.

The original water content means the water present in the concrete mix at the time of setting.

The procedure involves the determination of:

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

b) Combined water still present in the prepared concrete sample as cement hydrates.

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

4.1 Capillary porosity

Prepare the sample as described in **2.3** and dry at 105 ± 5 °C for not less than 16 h and cool in a desiccator.

Weigh the dried slice and immerse in carbon tetrachloride*. The carbon tetrachloride is contained in a vessel that can be evacuated for example, a 250-mm diameter vacuum desiccator. Reduce the pressure in the vessel to less than 13.3 kPa by means of an efficient water pump or vacuum pump**.

The air from the capillaries in the concrete is evolved, rapidly at first and then more slowly. The removal of the capillary air is complete if no further air is released even when the evacuated vessel is cautiously given a sharp tap. The time required to remove the air may be several hours. *Boiling* of the carbon tetrachloride induced by low pressure with

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* *Avoid breathing the vapour and contact of the liquid with the skin and eyes is strongly recommended that polythene gloves should be used when handling samples.*

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

continued evacuation should not be confused with air evolution and is normally recognizable by the tendency of large bubbles to appear at a few points on the concrete surface.

When the air evolution has ceased, the vacuum is released and the sample kept immersed at atmospheric pressure for a further 5 minutes.

Remove the sample from the carbon tetrachloride. Allow excess superficial liquid to drain away and quickly wipe the sample dry. Without delay, place the saturated sample in a weighed polythene (polyethylene) bag, seal to prevent loss of liquid by evaporation, and weigh.

Calculate the mass of carbon tetrachloride required to fill the pores of the concrete and from this mass derive the equivalent weight of water assuming that the density of the carbon tetrachloride is 1.6 g/ml.

$$\%\ \text{capillary water} = \frac{\text{mass of carbon tetrachloride absorbed}}{1.6 \times \text{mass of dried sample}} \times 100$$

4.2 Combined water and cement content

Remove the slice from the polythene (polyethylene) bag, (If test No. 3, for bulk density, is required, weigh the saturated slice suspended in carbon tetrachloride see 5). Heat the sample to constant mass at 100 °C to 105 °C. Crush the whole sample to pass a 150-µm *CS fine mesh test sieve and determine combined water and cement content.

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* *CS 124 Test sieves.*

4.2.1 *Combined water*

The combined water is determined directly by igniting a powdered sample at 1000 °C in a stream of dried air or nitrogen and weighing the evolved water after absorption on *dried* magnesium perchlorate (anhydrous).

4.2.1.1 Apparatus

The apparatus consists of a small absorption vessel containing *dried* magnesium perchlorate, through which the gas is passed before entering a silica combustion tube that is heated in a furnace capable of reaching 1000 °C (bright red heat). The exit from the combustion tube should be packed with silver wool. If silicone rubber connections are used it is advisable to protect them with heat reflectors. The gas leaving the combustion tube is passed through two weighable absorption tubes. The first quarter of each absorption tube is packed with silica gel and the remainder with *dried* magnesium perchlorate. A bubbler containing sulphuric acid is fitted after the exit end of the second absorption tube to allow the gas flow to be observed (Fig. 1 shows one method of achieving these requirements).

4.2.1.2 Procedure

4.2.1.2 a) 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.

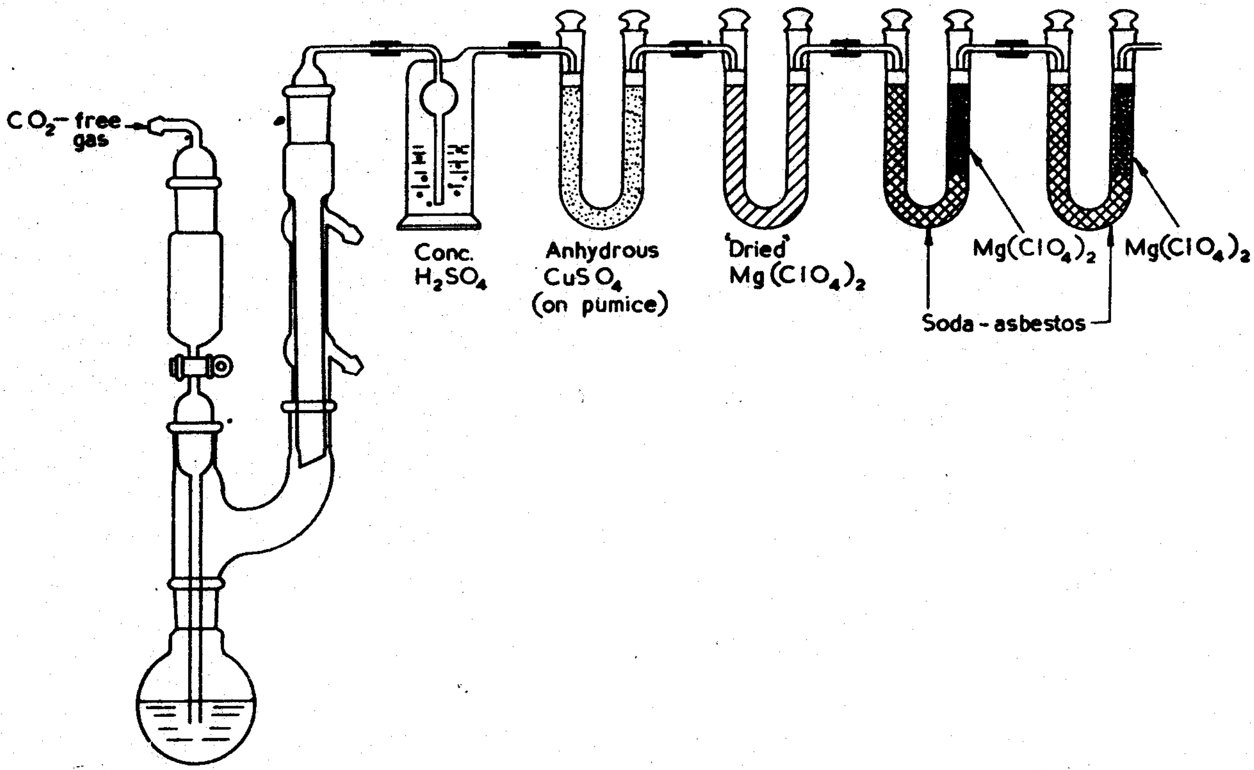

FIGURE 1 - An apparatus for determination of CO_2

4.2.1.2 b) When the combustion tube has cooled to below 100 °C, weigh the absorption tubes and fit into the train. A quantity of approximately one gram of the sample is weighed to an accuracy of one tenth of a milligram in 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 three 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 air for a further 15 min. Disconnect the absorption tubes, transfer to the balance case and weigh after 15 min.

4.2.1.2 c) To determine the *blank* of the apparatus which should be less than one milligram, carry out the procedure from the beginning of **4.2.1.2.b**, with no sample in the combustion boat.

4.2.1.3 Calculation

$$\text{\% combined water} = \frac{\text{gain in mass of absorption tubes} - \textit{blank}}{\text{mass of sample}} \times 100$$

4.2.2 *Cement content*

Determine the cement content of the prepared sample by the method that has been used for the main analysis or by the method which would have been used, depending on the aggregate type, for the main determination of cement content.

4.2.3 *Corrections for porosity and combined water of the aggregate*

If a separate sample of aggregate is not available for the determination of its porosity and combined water content, separate the coarse aggregate from the reserved portion of the concrete sample (see 2.3). Make sure that no work requiring the use of the intact sub-sample (for example 5.1) remains to be done. Then break down this reserved sample, avoiding, as far as possible fracture of the aggregate, and separate into coarse and fine fractions on a 4.75-mm CS fine mesh test sieve (Do not heat to assist break down of the matrix). Clean the aggregate related on the sieve by chipping with a chiseledged tool. Ensure complete removal of adhering cement matrix; for natural aggregates essentially insoluble in dilute hydrochloric acid this can be achieved by washing in 10 per cent hydrochloric acid; with other types it is necessary to use mechanical abrasion even if some loss of the aggregate surface occurs. Wash, and dry the cleaned aggregate at 105 ± 5 °C and determine its porosity and combined water content.

4.2.3.1 Aggregate porosity

Weigh the dried aggregate and determine the capillary porosity by the method given in 4.1. For convenience, enclose the aggregate in a plastic mesh bag or support on a sieve. Dry the surface of the aggregate before re-weighing by shaking off the excess liquid and rapidly rubbing in cloth.

$$\%\text{ capillary porosity} = \frac{\text{mass of carbon tetrachloride absorbed}}{1.6 \times \text{mass of dry coarse aggregate}} \times 100$$

4.2.3.2 Aggregate combined water

Heat the saturated aggregate to constant mass at 105 ± 5 °C and crush to pass a 150-µm CS fine mesh test sieve. Determine the combined water by the method given in 4.2.1.2.

4.3 Calculation

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

Let determined capillary water of slice	= Q %;
Let determined capillary porosity of aggregate	= q %;
Let combined water of concrete	= X %;
Let combined water of aggregate	= Y %;
Assume combined water of cement	= 1.0 %;
Let cement content	= C %;
Let aggregate content	= R %;

Then corrected capillary water $= Q - \dfrac{qR}{100}$ %;

Corrected combined water $= X - \dfrac{YR}{100} - \dfrac{1.0C}{100}$ %;

It is assumed that none of the water of hydration has been replaced by carbon dioxide.

$$\%\ \text{original water} = \%\ \text{corrected capillary water} + \%\ \text{corrected combined water.}$$

$$\text{Water/cement ratio} = \frac{\%\ \text{original water}}{\%\ \text{cement}} = \frac{Q + X - \frac{qR}{100} - \frac{YR}{100} - \frac{1.0C}{100}}{C}$$

5 BULK DENSITY

Test No. 3 - Test for bulk density

The determination of the bulk densities of the concrete, saturated with water and dried at 105 °C, is covered in Part 2 of this standard. The method there described is applicable to 100 mm or 150 mm cubes or other specimens of similar size; there specimens are used for each determination.

Such specimens may bot be available for the concrete to be analysed, but in this connection the dry density is usually required solely for the conversion of mass percentages to kilogrames per cubic metre. Therefore the reserved portion (see 3.3.2) or the largest single piece thereof, may be used, before beginning any work involving its destruction (for example 4.2.3).

5.1 Immerse a single sound piece of the concrete in water for 30 min or more (complete saturation is unnecessary), and weigh it suspended in water (mass *Wc*). Quickly mop surplus water from the surface and re-weigh in air (mass *Wb*). Dry the concrete at 105 °C for three days (this time may be reduced when the specimen is much smaller than a 150-mm cube), cool to near room temperature in a closed vessel such as a desiccator and re-weigh in air (mass *Wa*).

Then dry bulk density $= \dfrac{\rho \times Wa}{Wb - Wc} \times 1000$ kg/m^3

Where ρ is the density of water at the operating temperature in gm/cm^3.

5.2 Alternatively, if the capillary porosity has been determined by the method given in 4.1, bulk density can be derived by weighing the saturated slice while suspended in carbon tetrachloride (see 4.2). The above equation is used, ρ now being the density of carbon tetrachloride at the operating temperature.

6 TYPE OF CEMENT

Test No. 4 - Test for type of cement

Differentiation between various types of cement present in a concrete, either alone or in admixture, 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. It is strongly advisable to supplement results thus obtained by microscopic examination of a polished specimen containing cement particles still unhydrated.

6.1 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 (suing, say, woven wire cloth of 90-µm nominal aperture size). If insufficient sample is thus obtained, further very careful breakdown is necessary.

Analyse the very fine material so obtained for, say silicon dioxide, calcium oxide, aluminium oxide, ferric oxide, magnesium oxide, sulphur trioxide by suitable methods. Compare with typical analyse of various types of cement.

6.2 Microscopic examination

Microscopic examination should be left to an experienced microscopist. Select a small solid piece of the concrete (about 20 mm cube) preferably not containing any large aggregate and not having a finished surface, and dry for 12 h at 105 °C. Embed in epoxy resin by casting under vacuum into a plastic tube and cure at the recommended temperature. Cut through the concrete with a diamond saw and grind the cut surfaces with 30-µm carboroundum powder lubricated with kerosene. Continue grinding with 13-µm carboroundum powder. Remove all traces of grinding powder and carefully polish with progressively finer diamond compound on a paper surfaced lead, lap, being careful not to pluck cement particles out of the surface. After the final polish with 0.25-µm diamond powder, clean the surface and pat dry with tissue.

Examination of the polished surfaces of the concrete section by reflected light microscopy will reveal coarse particles of unhydrated cement clinker. After suitable etching, these particles show the phases characteristic of the type of cement. In cases of doubt, it is advisable to prepare specimens with known cements for comparison.

7 TYPE OF AGGREGATE USED

Test No. 5 - Test for type of aggregate used

Prepare a cut slice and examine the sections of aggregate particles so exposed by visual, chemical or physical means. Refer to a petrologist if other than a general classification is necessary.

8 MISCELLANEOUS

Test for chloride, sulphate and sulpho-aluminate

In general, the ground sub-sample (see **2.3.4**) can be used for all these tests. However, since the determinations are always related to cement content, a sample in which the cement matrix has been concentrated, that is: the fine fraction obtained in the tests described in **3.2**, presents some advantages. Whichever sample is used, a knowledge of its cement content will be necessary.

8.1 Chloride content

Use the method described below.

8.1.1 *Procedure*

Weigh into a stoppered conical falsk a quantity of sample expected to be equivalent to about two grams cement. Disperse with 25 ml of water and add 10 ml of nitric acid (d = 1.42). Add 50 ml of water, heat to near boiling and keep warm for 10 min to 15 min (If the supernatant liquid is turbid, filter through a rapid paper* and wash with hot water). Cool to room temperature and add a measured excess of standard 0.1 N silver nitrate (16.989 g pure dry $AgNO_3$ in one litre).

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* *Whatman No. 541 or equivalent is suitable.*

Add 2 ml to 3 ml nitrobenzene, stopper the flask and shake vigorously to coagulate the precipitate. Add one millilitre of ferric indicator solution (100 ml cold saturated solution of Ammonium ferric sulphate plus 10 ml of nitric acid (d = 1.42) and titrate with standard 0.1N ammonium thiocyanate to the first permanent red colour.

Standardize the ammonium thiocyanate (7.6 g NH_4CNS per litre) against the silver nitrate by the same method as above.

8.1.2 *Calculation*

Report as anhydrous calcium chloride per cent of cement content.

$$CaCl_2 \text{ \% of cement content} = \left[V - \frac{tX}{0.1}\right] \frac{0.555}{m} \times \frac{100}{C}$$

where

m = mass of sample, in grams ;

V = volume of 0.1N $AgNO_3$ added in millilitres ;

t = titre of NH_4CNS solution ;

X = normality of NH_4CNS solution ; and

C = cement content % of sample.

NOTE - Although usually reported as calcium chloride, the chloride may in fact be present in other forms, for example, sodium chloride.

8.2 Sulphate content

8.2.1 *Procedure*

Weigh into a 400-ml beaker a quantity of sample expected to be equivalent to about one gram cement. Disperse with 25 ml of water and add 10 ml of hydrochloric acid (d = 1.18). If effervescence is considerable, immediately cover. Add 50 ml of hot water, cover and boil gently for 5 min to 10 min. Filter through a retentive paper*, washing the residue thoroughly with hot dilute hydrochloric acid (1 +99). Heat the filtrate to boiling and just neuralize to methyl red with dilute ammonia solution (1 + 1).

Immediately add one millilitre hydrochloric acid (d = 1.18) and then add dropwise to the boiling solution 10 ml of barium chloride solution (100 g/l). Boil gently for 5 min, keep hot for 30 min and allow to cool for a minimum of one hour. Filter on a slow paper* 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 for 30 min, cool and weigh.

8.2.2 *Calculation*

$$SO_3\ \% = \frac{\text{mass of precipitate} \times 34.3}{m}$$

where

m = mass of sample, in grams

NOTE - Sulphur trioxide in unattacked Portland cement concrete should be not much greater than 3 per cent of cement content.

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* *Whatman No. 40 or equivalent is suitable.*

8.3 Sulpho-aluminate content

Knowledge of the distribution of Sulphur trioxide between sulpho-aluminate and gypsum may be required to determine the conditions in which sulphate attack has taken place. Sulpho-aluminate is stable only in the alkaline conditions normal in concrete, being converted to gypsum by acid; it is thus possible to distinguish between for example, attack by ground water or by the products of sulphur oxidizing bacteria.

The principle of the test is that sulpho-aluminate is not extracted by lime water whereas the other sulphates present are soluble.

8.3.1 *Procedure*

Prepare a lime suspension by shaking 2.5 g calcium oxide (obtained by heating 4.5 g pure calcium carbonate at 1000 °C for 60 min) with one litre distilled water. Store in an airtight polyethylene bottle and shake thoroughly immediately before use.

Weigh a quantity of sample thought to be equivalent to about 0.5 g cement into a stoppered conical flask. Add 50 ml of lime suspension and shake mechanically for 10 min. Filter through retentive paper* and collect the filtrate in a 400-ml beaker. Return the paper and residue to the flask and add a further 50 ml of lime suspension. Macerate the paper and shake for 30 min. Allow to stand overnight. Shake for a further 2 h to 3 h and then filter through a retentive paper* into the 400-ml beaker. Wash the flask and filter 5 times with 25 ml portions of the lime suspension. Neutralize the combined filtrate and washings to methyl red with hydrochloric acid (d = 1.18) and add one millilitre in excess. Complete the determination of sulphate in this solution as given in **8.2.1**.

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* *Whatman No. 40 or equivalent.*

8.3.2 *Calculation*

Calculate % lime soluble SO_3 as given in **8.2.2** then,

$$\% \text{ sulpho-aluminate expressed as } SO_3 = \% \text{ total } SO_3 - \% \text{ lime soluble } SO_3$$

9 REPORT

Report the following information for each sample:

a) identification marks and other relevant details supplied with the sample;

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

c) any assumptions made in the analysis (for example, type of aggregate, aggregate analysis);

d) results obtained by applying the tests required, together with magnitude of probable errors; and

e) conclusions regarding the concrete quality where required.

Report separately the following supplementary information:

a) any other results and observations obtained coincidentally to the tests required; and

b) the results and observations of any other tests done at the analyst's discretion.

APPENDIX A

SPECIMEN FORM FOR COLLECTING SAMPLES OF MORTAR CONCRETE

1 Type of structure :

2 Date of commencement :

3 Date of completion of work :

4 Date of collecting samples :

5 No. of samples taken :

6 Identification No. :

7 Position of samples taken with a suitable sketch (if relevant) :

8 Representativeness of sampling (if relevant) :

9 Nature of testing required :

10 Specimen of seal (if relevant) :

11 Collected by (signature, name of officer with designation, class or grade) :

12 Collected in the presence of :
(Signature, name and designation)

1

2

3

SLS CERTIFICATION MARK

The Sri Lanka Standards Institution is the owner of the registered certification mark shown below. Beneath the mark, the number of the Sri Lanka Standard relevant to the product is indicated. This mark may be used only by those who have obtained permits under the SLS certification marks scheme. The presence of this mark on or in relation to a product conveys the assurance that they have been produced to comply with the requirements of the relevant Sri Lanka Standard under a well designed system of quality control inspection and testing operated by the manufacturer and supervised by the SLSI *which includes surveillance inspection of the factory, testing of both factory and market samples.*

Further particulars of the terms and conditions of the permit may be obtained from the Sri *Lanka Standards Institution, 17, Victoria Place, Elvitigala Mawatha, Colombo 08.*

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The principal objects of the Institution as set out in the Act are to prepare standards and promote their adoption, to provide facilities for examination and testing of products, to operate a Certification Marks Scheme, to certify the quality of products meant for local consumption or exports and to promote standardization and quality control by educational, consultancy and research activity.

The Institution is financed by Government grants, and by the income from the sale of its publications and other services offered for Industry and Business Sector. Financial and administrative control is vested in a Council appointed in accordance with the provisions of the Act.

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All members of the Technical and Sectoral Committees render their services in an honorary capacity. In this process the Institution endeavours to ensure adequate representation of all view points.

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