

Construction Standard

CS1:2010

Testing Concrete

Volume 2 of 2

香港特別行政區政府

The Government of the Hong Kong Special Administrative Region

Construction Standard CS1:2010

Volume 2 of 2

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The Government of the Hong Kong Special Administrative Region

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FOREWORD

This Construction Standard has been prepared by the Working Group on Review of CS1 under the Standing Committee on Concrete Technology, Development Bureau, the Government of the Hong Kong Special Administrative Region. This Construction Standard supersedes CS1:1990. It sets out methods and procedures to be adopted for sampling and testing concrete both on site and in the laboratory.

The contents of this Construction Standard are based primarily on the following British Standards (BS), European Standards adopted as British Standards (BS EN), International Organization for Standardization (ISO) Standards, Reunion Internationale des Laboratoires et Experts des Materiaux, systemes de construction et ouvrages (RILEM), (International Union of Laboratories and Experts in Construction Materials, Systems, and Structures) and ASTM International Standards, with modifications to suit local conditions and practices.

BS 1881:Part 124:1988
 BS 1881:Part 103:1993
 BS EN 12350-1:2009
 BS EN 12350-2:2009
 BS EN 12350-3:2009
 BS EN 12350-5:2009
 BS EN 12350-6:2009
 BS EN 12350-7:2009 Amd No. 1/2013
 BS EN 12390-1:2000
 BS EN 12390-2:2009
 BS EN 12390-3:2009 Amd No. 1/2013
 BS EN 12390-4:2000
 BS EN 12390-5:2009
 BS EN 12390-6:2009 Amd No. 1/2013
 BS EN 12390-7:2009
 BS EN 12390-8:2009 Amd No. 1/2013
 BS EN 12504-1:2009
 BS EN 13294:2002
 ISO 1920-2:2005(E)
 RILEM TC 106-2:2000
 RILEM TC 106-3:2000 Amd No. 1/2013
 ASTM C1202-97

This Construction Standard comprises two volumes. Volume 1 generally covers site operations, including methods of sampling fresh concrete, testing of fresh concrete and making and curing test specimens. Volume 2 is intended to be used by laboratory staff and covers sampling of fresh concrete in the laboratory, methods of determining the physical properties of hardened concrete and the sampling and chemical analysis of hardened concrete.

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The contribution from the following organizations on the drafting of the Construction Standard is gratefully acknowledged:

- The Hong Kong Institution of Engineers
- The Concrete Producers Association of Hong Kong Ltd.
- The Association of Construction Materials Laboratories Ltd.
- The Hong Kong Construction Association Ltd.
- The Mass Transit Railway Corporation Ltd.

The electronic files of the Construction Standard including amendment, if any, can be found on the website of the Civil Engineering and Development Department.

September 2010

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GENERAL NOTES

The following notes are relevant, where applicable, to all the sections of this Standard.

1. Reports

- (a) It is a mandatory requirement for all reports to contain the name and signature of the person responsible for the sampling or the test. The person responsible for the test is not necessarily the same as the person actually carrying out the test. For HOKLAS accredited laboratories, the person responsible should be an approved signatory.
- (b) The person responsible for the test must ensure that all the information listed for the inclusion in a Report is fully and accurately stated. However, some of the information listed for inclusion in a Report may not be known to the person responsible for the test. In such a case the words 'not known' should be entered in the Report.
- (c) If any test is performed on a specimen which does not fully comply with this Standard (e.g. a cube which has not been cured in accordance with Section 10) or if the test itself does not fully conform to the requirements of this Standard, the relevant details must be stated in the Report.

2. Apparatus

In general, the lists of required apparatus in this Standard do not include standard items of equipment and consumables which are normally found in a well equipped testing facility.

3. Tolerances

The tolerances stated in various sections of this Standard shall be interpreted as follows:

- (a) **Flatness.** The surface specified as having a flatness tolerance shall lie between two parallel planes. The perpendicular distance between the planes is the flatness tolerance quoted. The flatness can be assessed by the measurement of straightness in various positions of a plane surface.
- (b) **Perpendicularity.** Where a surface is specified as having a perpendicularity tolerance relative to another surface, it shall lie between two parallel planes perpendicular to the reference surface. The perpendicular distance between the planes is the perpendicularity tolerance quoted. Where a surface is specified as having a perpendicularity tolerance relative to a datum line (e.g. the axis of a cylinder), it shall lie between parallel planes perpendicular to the datum line. The perpendicular distance between the two planes shall be the perpendicularity tolerance quoted.
- (c) **Parallelism.** Where a surface is specified as having a parallelism tolerance relative to another surface, it shall lie between two planes parallel to the reference surface. The perpendicular distance between the planes shall be the parallelism tolerance quoted.
- (d) **Straightness.** The line specified as having a straightness tolerance shall lie between two parallel lines. The perpendicular distance between the lines is the straightness tolerance quoted.

4. Singular and plural

Words importing the singular only also include the plural and vice versa where the content requires.

SECTION 11

MIXING AND SAMPLING FRESH CONCRETE IN THE LABORATORY

11.1 SCOPE

This Section describes the methods for the preparation of materials and the batching, mixing and sampling of concrete in the laboratory.

11.2 DEFINITIONS

Batch is the quantity of concrete mixed in one cycle of operations of a mixer or the quantity mixed by hand in one cycle of operations.

11.3 APPARATUS

The following apparatus is required:

- (a) Mixer for laboratory use (see C1. C1.1).
- (b) Sample container (see C1. C2.1).
- (c) Sample tray (see C1. C3).
- (d) Scoop (see C1. C4).
- (e) Square-mouthed shovel.

Further details of the apparatus are given in Appendix C.

11.4 CONSTITUENT MATERIALS

11.4.1 Sampling

If bulk samples are supplied, subsamples for the test portions shall be taken in accordance with the relevant Standard and shall be representative of the bulk materials. Where required, samples shall be accompanied by a certificate of sampling in accordance with the relevant Standard. Otherwise, the materials in the proportions supplied or specified for the mix shall be used.

11.4.2 Cement, pulverized-fuel ash (PFA) and other cementitious materials

Upon their arrival at the laboratory, the materials shall be stored in separate airtight containers of appropriate size in a dry place. Before use, each separate material shall be stirred thoroughly either with a hand tool or in a

suitable mixer, in such a manner as to ensure the greatest possible uniformity. Care shall be taken to avoid the intrusion of foreign matter or loss of material.

11.4.3 Aggregates

The moisture content (as a percentage of the oven dry mass) of the aggregates shall be determined in accordance with BS 812:Part 109.

The aggregates shall be in one of the following conditions:

- (a) Oven dried as described in BS 812:Part 100.
- (b) Air dried at (25 ± 5) °C.
- (c) Saturated surface dry as described in BS 812:Part 100.
- (d) Saturated by soaking in water for at least 24 hours.

The aggregate for each batch shall be used in separate size fractions. Care shall be taken to prevent loss of fines during the saturation process.

NOTE. *The easiest way to standardise the condition of aggregate is by saturation.*

11.4.4 Temperature

All materials shall be at a temperature of (25 ± 5) °C before mixing.

11.5 BATCHING

The quantity of concrete in each batch shall be at least 10 % more than that required for the proposed test.

For each batch, the cement, PFA, other cementitious materials, aggregate and water shall be weighed to an accuracy of 0.5 % or better.

If an admixture is to be incorporated, the amount shall be measured to within 0.5 % of the specified dosage and the manufacturer's instructions for use shall be followed.

11.6 MIXING

11.6.1 General

The concrete shall be mixed in a room having an ambient temperature of $(25 \pm 5)^\circ\text{C}$ and a relative humidity of not less than 50 %.

If the aggregate is dry, it shall be soaked with some of the mixing water before adding other materials. Loss of water by evaporation shall be avoided.

After the dry aggregate has been allowed to soak, admixtures if required may be added together with the remaining portion of the mixing water, except in the case of admixtures that need to be added shortly before using the concrete.

The concrete shall be mixed, preferably by machine or alternatively by hand, in such a manner as to avoid loss of water or other materials.

NOTE. *Different methods of mixing may lead to different results.*

11.6.2 Machine mixing

The size of the batch shall be of sufficient volume to allow thorough mixing.

Before using the mixer, any fresh concrete remaining from a previous batch shall be cleaned out. No free water shall remain in the mixer. If the mixer is dry it shall be wiped with a damp cloth. When using a tilting drum mixer, a small preliminary batch, of similar proportions to the main batch, shall be mixed and discharged immediately before the main batch in order to coat the mixer.

The mixer shall be charged with about one half of the coarse aggregate, then with the fine aggregate, then with the cement and finally with the remaining coarse aggregate. All the water shall be added during the first 30 seconds of mixing. Mixing shall continue after all the materials have been added for at least 2 minutes until the concrete appears to be uniform and homogeneous. When using a pan mixer not fitted with a discharging gate the concrete shall be heaped together before sampling. When using other types of mixer the discharged

concrete shall be remixed on a sample tray in such a manner as to ensure uniformity.

11.6.3 Hand mixing

The concrete batch shall be mixed on a sample tray with a square-mouthed shovel or similar suitable equipment Amd No. 1/2013, using the following procedure:

The cement and fine aggregate shall be mixed dry until the mixture is uniform. The coarse aggregate shall be added and mixed dry with the cement and fine aggregate until the coarse aggregate is uniformly distributed throughout the batch. The water shall then be added and the whole mixed for at least three minutes and until the concrete appears to be homogeneous.

11.7 SAMPLING AND TESTING THE CONCRETE

Tests on fresh concrete and the preparation of specimens for tests on hardened concrete shall start as soon as possible after completion of mixing. The required operations shall be carried out during a period of not more than one hour from the addition of the water to the cement. The fresh concrete shall be protected against gaining or losing water.

Provided that care is taken to ensure that no water or other material is lost, the concrete used in workability and density tests may be remixed with the remainder of the batch before making any specimen for testing hardened concrete. The period of remixing should be as short as possible but sufficient to produce a homogeneous mass.

11.8 REPORT

The report shall affirm that the preparation of materials, batching, mixing and sampling were carried out in accordance with this Standard. The report shall Amd No. 1/2013 include the following:

- Name and address of laboratory.
- Name of project, if any.
- Batch identity number.
- Date and time of adding water to the mix.
- Description of materials, including moisture

- (f) content and condition of the aggregates.
- (g) Particle size of aggregates.
- (h) Grade of concrete.
- (i) Actual quantities or proportions of material batched and admixtures added.
- (j) Method of mixing, type and rated capacity of mixer.
- (k) Any observations on the appearance of the fresh concrete, e.g. segregation and bleeding.
- (l) Name and signature of person responsible for mixing and sampling.

SECTION 12

DETERMINATION OF COMPRESSIVE STRENGTH OF CONCRETE CUBES

12.1 SCOPE

This Section describes the method of determining the compressive strength of concrete cubes.

12.2 APPARATUS

The following apparatus is required:

- (a) Compression testing machine (see C1. C5).
- (b) Cube checking jig (see C1. C6).
- (c) Calliper (see C1. C7).
- (d) Weighing equipment of 20 kg capacity (see C1. C8.1).
- (e) Feeler gauges (see C1. C9).

Further details of the apparatus are given in Appendix C.

12.3 PROCEDURE

12.3.1 Preparation

Cubes stored in water or a mist chamber shall be tested within one hour of removal from the water or mist chamber, whilst they are still wet. Surface water and grit on the cube shall be wiped off and projecting fins removed.

12.3.2 Measurement of cubes

All cubes shall be visually inspected. Any cube which was unprotected on delivery or has unsatisfactory features shall be recorded as such. A cube having an edge broken for 20 mm or more in any direction shall be recorded as 'a cube with damaged edge'.

All cubes shall be checked to ensure that they satisfy the perpendicularity requirement given in Section 7 of this Standard. If the cube checking jig is used for this purpose, the procedure described below shall be followed.

Each cube shall be placed in the cube checking jig with the trowelled surface upwards, and the contact between the cube and jig shall be checked with the feeler gauges. The cube shall then be turned through 90° and the check

repeated. Should the perpendicularity of the cube not be within 1 % of the cube dimension, the amount of out of perpendicularity shall be recorded and the cube described as irregular.

The cube dimensions between the centres of the three pairs of opposing faces shall be measured with a calliper or other means which provide the same accuracy of measurement and recorded to the nearest 0.1 millimetre. A cube whose dimensions differ from the nominal dimensions by more than 1 mm on width or 2 mm on height as cast shall be recorded as 'oversize' or 'undersize' as appropriate.

Each cube shall be weighed and the as-received or saturated density determined in accordance with Section 16 of this Standard.

12.3.3 Placing the cube in the testing machine

The bearing surfaces of the testing machine shall be wiped clean and any loose grit or other extraneous material shall be removed from the surfaces of the cube which are to be in contact with the platens. No packing other than the auxiliary platens shall be used between the cube and machine platens. The cube shall be carefully centred on the lower platen. The trowelled surface shall be vertical.

12.3.4 Loading

The load shall be applied steadily and without shock such that the stress is increased at a rate within the range (0.6 ± 0.2) MPa/s until no greater load can be sustained. The maximum load applied to the cube shall be recorded.

12.4 TYPE OF FAILURE

Any unusual features in the type of failure shall be recorded. Examples of satisfactory failures and of some unsatisfactory failures as shown in Figure 13. The assessment of type of failure is not applicable to cement grout cubes.

12.5 CALCULATION AND EXPRESSION OF RESULTS

The cross-sectional area of the cube shall be calculated from the measured dimensions. The compressive strength of the cube shall be calculated by dividing the maximum load by the cross-sectional area. The result shall be expressed to the nearest 0.1 MPa. The density of the specimen shall be calculated using the measured dimensions or the volume obtained from the water displacement method.

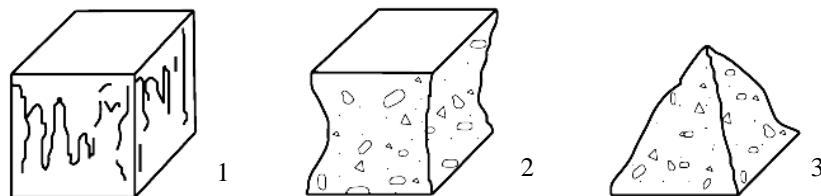
12.6 TEST REPORT

The report shall affirm that the test was carried out in accordance with this Standard and shall include the following:

- (a) Identification number of the specimen.
- (b) Date of receipt of the cube at the laboratory.
- (c) Date of test.

- (d) Age of the specimen at test.
- (e) Condition of the specimen when received (including poor compaction, honeycombing, incorrect dimensions or out of perpendicularity).
- (f) Curing report, if cured in laboratory.
- (g) Measured dimensions of specimen.
- (h) Mass of the specimen (as-received or saturated).
- (i) Density of the specimen (as-received or saturated, and the method of determining the volume).
- (j) Maximum load at failure.
- (k) Compressive strength.
- (l) Appearance of the concrete and type of fracture if these are unusual.
- (m) Other remarks if any.
- (n) Name and signature of person responsible for the test.

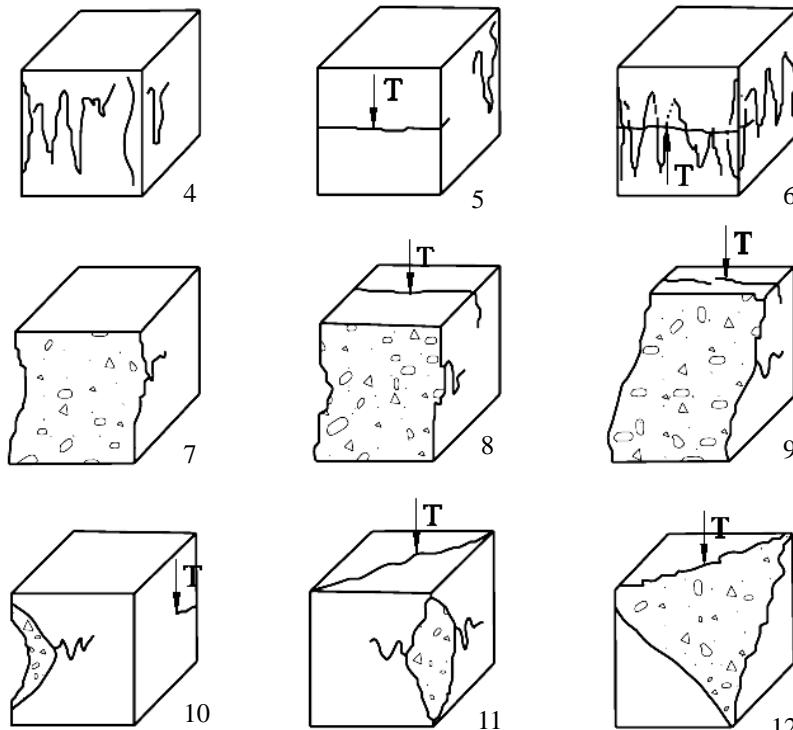
NOTE. *The reports on the production and curing of the test specimen (see Sections 7 and 10) may conveniently be added to this report.*



Explosive Failures

Note. All four exposed faces are cracked approximately equally, generally with little damage to faces in contact with the platens.

Satisfactory Failures



Note. T = tensile crack

Unsatisfactory Failures

Figure 13 - Failure Patterns of Cubes

SECTION 13

DETERMINATION OF TENSILE SPLITTING STRENGTH

13.1 SCOPE

This Section describes the method of determining the tensile splitting strength of cylindrical concrete test specimens.

13.2 DEFINITIONS

Tensile splitting strength is the theoretical maximum indirect tensile stress obtained by splitting the specimen under a concentrated compressive line load.

13.3 APPARATUS

The following apparatus is required:

- (a) Compression testing machine (see C1. C5).
- (b) Jig for tensile splitting strength test (Figure 14) (see C1. C10).
- (c) Packing strips (see C1. C11).
- (d) Steel loading pieces (see C1. C12).

Further details of the apparatus are given in Appendix C.

13.4 TEST SPECIMENS

13.4.1 Specimen size

The test specimen shall be a 150 mm diameter by 300 mm long cylinder made, cured and stored in accordance with Sections 9 and 10 of this Standard. The perpendicularity and straightness on the load bearing surface of the specimen shall be checked to ensure it satisfies the tolerance requirements of Section 9.

13.4.2 Dimensions

The dimensions of each specimen shall be measured in accordance with Section 16 of this Standard.

13.4.3 Mass and density

Each specimen shall be weighed to the nearest

10g and the as-received or saturated density determined in accordance with Section 16 of this Standard.

13.5 PROCEDURE

Mist chamber or water cured specimens shall be tested within one hour of removal from the water or chamber, whilst they are still wet.

13.5.1 Preparation for testing

The bearing surfaces of the testing machine, the steel loading pieces and the packing strips shall be wiped clean; any loose material on the surfaces of the test specimen which are to be in contact with the packing strips shall be removed.

13.5.2 Positioning of the test specimen

The test specimen shall be placed in the centring jig with packing strips and loading pieces carefully positioned along the top and bottom of the plane of loading of the specimen. The jig shall then be placed in the machine so that the specimen is located centrally. The upper platen shall be parallel to the lower platen (see Figure 14).

13.5.3 Loading

The initial load applied to the test specimen shall not exceed 20% of the failure load. After application of the initial load, the load shall be applied Amd No. 1/2013 steadily and without shock such that the stress is increased at a rate within the range of 0.04 MPa/s to 0.06 MPa/s. Once adjusted, the rate shall be maintained at $\pm 10\%$ until failure. The maximum load applied to the specimen shall be recorded. The rate of increase of load may be calculated from the following expression:

$$s \times \frac{\pi}{2} \times L \times d \quad N/s \qquad \text{-----(13 - 1)}$$

where

- L is the average measured length of the specimen (in mm)
- d is the average measured diameter of the specimen (in mm)
- s is the increase in rate of stress, in MPa/s

13.6 CALCULATION AND EXPRESSION OF RESULTS

The tensile splitting strength f_{ct} in MPa is given by the equation:

$$f_{ct} = \frac{2F}{\pi \times L \times d} \quad \text{----- (13 - 2)}$$

where

- F is the maximum load (in N)
- L is the average measured length (in mm)
- d is the average measured diameter (in mm)

The tensile splitting strength shall be expressed to the nearest 0.05 MPa.

13.7 TEST REPORT

The report shall affirm that the test was carried out in accordance with this Standard and shall include the following:

- (a) Identification number of the specimen.
- (b) Date of receipt of the specimen at the laboratory.
- (c) Date of test.
- (d) Age of the specimen at test.
- (e) Condition of the specimen when received (include poor compaction, honeycombing or incorrect dimensions).
- (f) Curing report, if cured in laboratory.
- (g) Mass of the specimen (as-received or saturated).
- (h) Measured dimensions of the specimen.
- (i) Density of the specimen (as-received or saturated and the method of determining the volume).
- (j) Maximum load at failure.
- (k) Tensile splitting strength.
- (l) Appearance of concrete and type of fracture if these are unusual.
- (m) Other remarks if any.
- (n) Name and signature of person responsible for the test.

NOTE. The reports on the production and curing of the test specimen (see Sections 9 and 10) may conveniently be added to this report.

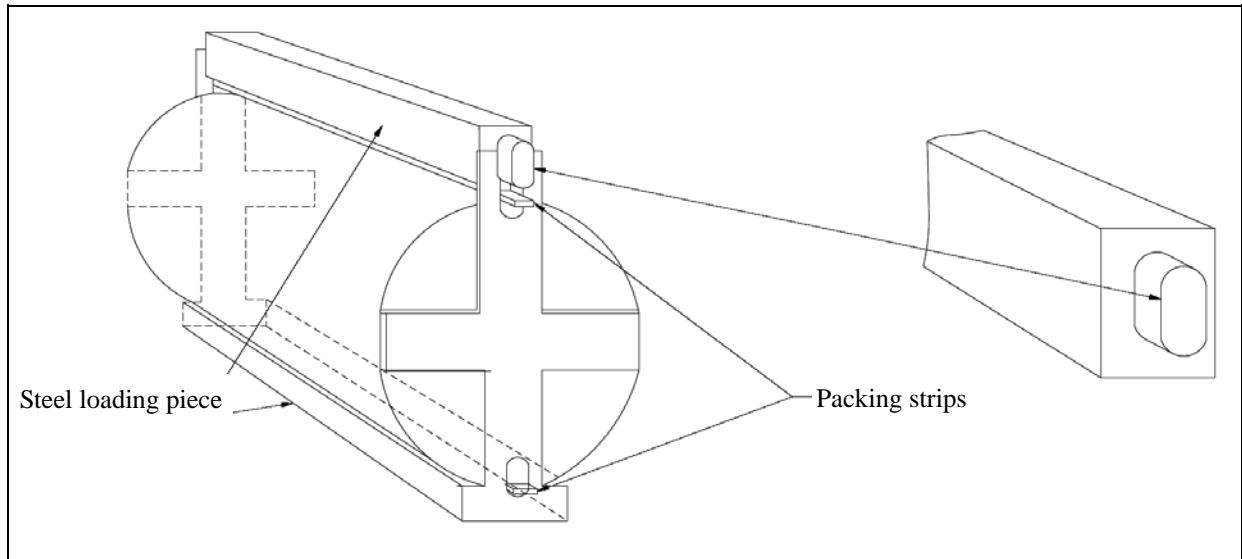


Figure 14 - Jig for Tensile Splitting Strength Test

SECTION 14

DETERMINATION OF FLEXURAL STRENGTH

14.1 SCOPE

This Section describes the method of determining the flexural strength of a test specimen of hardened concrete.

14.2 DEFINITIONS

Flexural strength is the theoretical maximum tensile stress reached in the bottom fibre of a test beam during a flexural strength test.

14.3 APPARATUS

The following apparatus is required:

- (a) Compression testing machine (see C1. C5).
- (b) Flexural loading device
(Figure 15) (see C1. C13).

Further details of the apparatus are given in Appendix C.

14.4 TEST SPECIMENS

14.4.1 Specimen size

Test specimens shall be 150 mm x 150 mm x 750 mm long moulded concrete beams made in accordance with Section 8 and cured in accordance with Section 10 of this Standard.

14.4.2 Tolerances

The specimen shall be checked with a try-square, straight edge and calliper to ensure that it satisfies the tolerance requirements of Section 8.

In addition, the flatness tolerance for the bearing surfaces at the four positions of the rollers shall be 0.2 mm.

14.4.3 Mass and density

The specimen shall be weighed to the nearest 10 g and the as-received or saturated density

determined in accordance with Section 16 of this Standard.

14.5 PROCEDURE

Specimens stored in water or a mist chamber shall be tested within one hour of removal from the water or mist chamber, whilst they are still wet.

The bearing surfaces of the supporting and loading rollers shall be wiped clean and loose grit or other extraneous material shall be removed from the specimen.

The test specimen shall be correctly centred in the machine with the trowelled surface vertical (see Figure 15). The rollers shall be placed at right angles to the longitudinal axis of the specimen.

There shall be no packing between the specimen and the rollers. The reference direction of loading shall be perpendicular to the direction of casting of the specimen. All loading and supporting rollers shall be in even contact with the test specimen before load is applied. After the application of the initial load, which does not exceed approximately 20 % of the failure load, test load shall be applied without shock such that the stress is increased continuously at a selected constant rate of $\pm 10\%$ within the range of 0.04 MPa/s to 0.06 MPa/s. Test load shall be applied until no greater load can be sustained.

Once adjusted, the rate of loading shall be maintained without change until failure occurs. The maximum load applied shall be recorded.

NOTE. A rate of increase of loading of 450 N/s is equivalent to an increase of stress of 0.06 MPa/s for a 150 mm specimen.

Failures outside the middle one-third of the distance between the supporting rollers shall render the test invalid.

14.6 CALCULATION AND EXPRESSION OF RESULTS

The flexural strength f_{cf} (in MPa) is given by the equation:

$$f_{cf} = \frac{450 \times F}{d_1 \times d_2^2} \quad \text{----- (14 - 1)}$$

where

F is the maximum load (in N)
 d_1 & d_2 are the width and depth of the specimen respectively (in mm) (see Figure 15).

The flexural strength shall be expressed to the nearest 0.1 MPa.

14.7 TEST REPORT

The report shall affirm that the test was carried out in accordance with this Standard and shall include the following:

- (a) Identification number of the specimen.
- (b) Date of receipt of the specimen.
- (c) Date of test.
- (d) Age of the specimen at the time of testing.
- (e) Condition of specimen when received (include poor compaction, honeycombing or incorrect dimensions).
- (f) Curing report, if cured in the laboratory.
- (g) Mass of the specimen (as-received or saturated).
- (h) Measured dimensions of the specimen.
- (i) Density of the specimen (as-received or saturated, and the method of determining the volume).
- (j) Rate of loading.
- (k) Maximum load at failure.
- (l) Flexural strength.
- (m) Appearance of concrete and type of fracture if these are unusual.
- (n) Other remarks if any.
- (o) Name and signature of person responsible for the test.

NOTE. The reports on the production and curing of the test specimen (see Sections 8 and 10) may conveniently be added to this report.

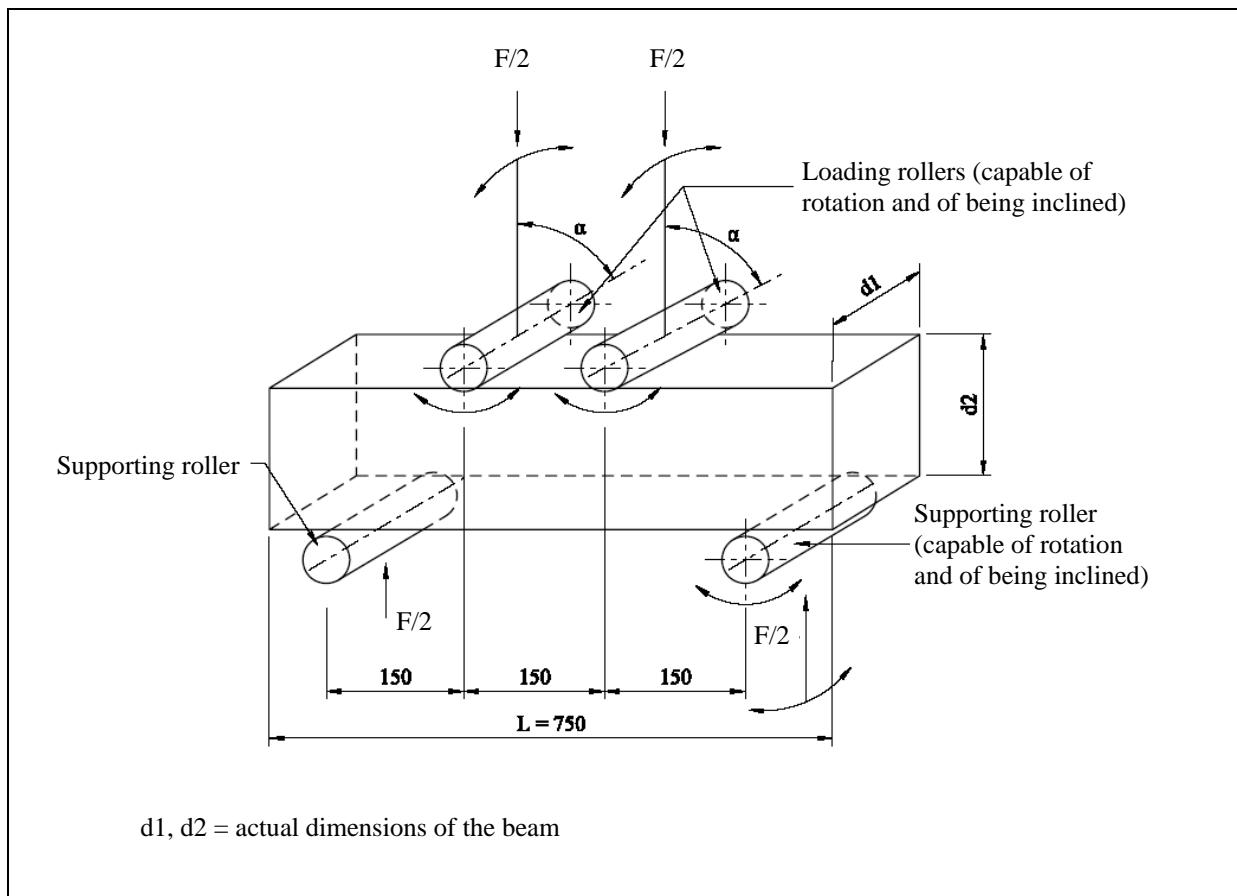


Figure 15 - Loading Arrangement of Flexural Strength Test

SECTION 15

OBTAINING CORE SAMPLES AND DETERMINATION OF THE COMPRESSIVE STRENGTH OF CONCRETE CORES

15.1 SCOPE

This Section describes the method of taking a core from concrete, preparing it for testing, determining its compressive strength and calculating the estimated in-situ cube strength.

NOTE. *Before deciding to drill cores for compressive testing, it is essential that full consideration be given to the necessity for the test, its aims and the value of the results which will be obtained. Reference should be made to specialist literature, such as Concrete Society Technical Report No. 11. It is recommended that before coring full agreement should be reached by all parties on the need for core testing and on how the results should be interpreted.*

15.2 APPARATUS

The following apparatus is required:

- (a) Compression testing machine (see C1. C5).
- (b) Coring machine (see C1. C14).
- (c) Grinding equipment for concrete cores (see C1. C15.1).
- (d) Steel plate (see C1. C16).

Further details of the apparatus are given in Appendix C.

15.3 TEST SPECIMENS

15.3.1 Size of cores

The test specimen shall preferably be of 100 mm diameter and in no case shall it be less than 75 mm diameter. The ratio of diameter to the maximum aggregate size shall be not less than 3. The length of core shall be sufficient to give the required length/diameter ratio after end preparation.

Particular care needs to be taken before using machines conforming to this Standard for compressive tests on small specimens, e.g. those with lateral dimensions significantly less than 90 mm. The main concern is that the ball-seating fitted to the upper platen may be too large to align satisfactorily on the top of such small specimens and special adaptations can be required. Another concern is the ability to

accurately determine the failure load of small or low strength specimens. Core specimens less than 90 mm diameter shall be tested by Class I compression testing machines only.

15.3.2 Drilling

Unless specifically required otherwise, cores shall be drilled perpendicular to the surface using a diamond core drilling bit and in such a manner as not to damage the cores. The drill shall be kept rigidly positioned during coring. Drilling through reinforcement shall be avoided wherever possible.

15.3.3 Identification

Immediately after cutting, each core shall be clearly and indelibly marked, indicating its location and orientation within the member. The direction of drilling relative to the direction of casting shall be recorded.

The core shall be marked to indicate distances in millimetres from the drilling surfaces so that the location in the element from which the test core came can be determined when the ends have been trimmed.

15.3.4 Testing machines

Testing machines shall either be Class I accurate to 1 % or Class II accurate to 2 %.

15.4 EXAMINATION OF CORES AFTER CUTTING

15.4.1 Compaction

Each core shall be examined for the presence of voids and cracks, and the position at which any honeycombing occurs shall be noted.

15.4.1.1 Voids

Upon request by the Engineer, an estimate of the excess voidage can be made by comparing the core sample against photographs.

It is possible to estimate the excess voidage of the core by comparing the number and size of the voids exposed on the drilled surface of the air-dry core with those displayed in Figures 16 (a) to 16 (e). These are 125 mm x 80 mm life-size photographs of the surface of the cores having known actual voidages, and hence inferable excess voidages, as shown in Table 4.

Table 4 - Assessment of Excess Voidage

| Figure No. | Excess Voids (%) |
|------------|------------------|
| 16 (a) | 0 |
| 16 (b) | 0.5 |
| 16 (c) | 1.5 |
| 16 (d) | 3.0 |
| 16 (e) | 13.0 |

The comparison of the surface voids of a given core with those shown in Figures 16 (a) to 16 (e) should always be made by two observers, in order to avoid extremes of subjective bias, care being taken to ensure that the voids are viewed in strong light angled to highlight them with shadows as in Figures 16 (a) to 16 (e). The recommended procedure for the comparison is as follows:

- cut a 125 mm x 80 mm rectangular aperture in a piece of thin card;
- place the card on the core with elastic bands;
- assess the excess voidage of the area of core in view by comparing it with Figures 16 (a) to 16 (e) and record the assessment;
- move the card to other areas and repeat the assessment until the cylindrical face of the core has been surveyed representatively;
- average the individual assessments and record the result to the nearest multiple of 0.5 %.

NOTE. Where the relative frequencies of small and large voids on the test core differ from those shown in Figures 16 (a) to 16 (e), estimation of the excess voidage may be facilitated by remembering that a void of a given diameter (or linear dimension) is equal in volume to eight voids having only half that diameter (or linear dimension).

NOTE. Where a photographic record of the air-dry core is required, the centre of the photograph should include that 125 mm x 80 mm area having an estimated excess

voidage nearest to the average for the whole core. The lighting should also be such that a photograph comparable in quality to Figures 16 (a) to 16 (e) is obtained, and the photograph should be reproduced to life size.

15.4.1.2 Honeycombing

Interconnected voids arising from, for example, inadequate compaction or lack of mortar.

15.4.2 Description of aggregate

When required, pieces of coarse aggregate shall be examined for general type, particle shape and size.

15.4.3 Distribution of materials

Each core shall be examined for evidence of segregation of the individual materials by visually comparing the approximate coarse aggregate/mortar ratio at different planes in the core.

15.4.4 Measurement of dimensions

The maximum and minimum lengths of the core as received shall be measured. The diameter and length of specimens cut from cores shall be measured with a calliper in accordance with Clause 16.6.1 of this Standard and the length of specimens shall be obtained before and after end preparation. The average length and diameter shall be calculated and the volume V_1 of the specimen (in m^3) obtained.

15.4.5 Measurement of mass and density

The mass and saturated density of each specimen shall be determined in accordance with Section 16 of this Standard before end preparation.

15.4.6 Measurement of reinforcement

The size and, if possible, spacing of any reinforcing bars shall be measured. The position of any reinforcement shall be determined by measuring from the centre of the exposed bars to the drilling surface as received and after end preparation. The accuracy of

measurement shall be to the nearest ± 1.0 mm.

15.4.7 Straightness

The straightness tolerance for the core shall be ± 3 % of the core diameter.

15.5 PREPARATION OF CORES

15.5.1 End preparation

For compressive strength testing the length/diameter ratio shall be between 1.0 and 1.2. For static modulus testing, the length/diameter ratio shall be between 2 and 5. When it is necessary to reduce the length of core, the core shall be sawn perpendicular to its longitudinal axis. Wherever possible, inclusion of reinforcement in the specimen shall be avoided.

Grinding is the preferred method of end preparation but if this is impracticable, the ends shall be capped in accordance with C1. 15.5.2. The ground or capped ends shall comply with the tolerances given in C1. 15.5.3 of this Standard.

15.5.2 Capping

Caps shall be made as thin as possible and shall not exceed 10 mm thickness at any point.

The capping material may be a suitable proprietary capping compound, a mixture of sulphur and fine siliceous sand or a mixture of sulphur and pulverized-fuel ash.

If a mixture of sulphur and fine siliceous sand is used, it shall consist of a mixture of equal parts by weight of sulphur and fine siliceous sand (most of which passes a 300 μm woven wire sieve and is retained on a 150 μm woven wire sieve) together with a small proportion (1 % to 2 %) of carbon black or a small proportion (2 % to 4 %) of polysulphide rubber. The mixture shall be heated to a temperature of 130 °C to 150 °C whilst being stirred continuously to ensure its homogeneity Amd No. 1/2013 and then allowed to cool slightly Amd No. 1/2013. The mixture shall be poured onto a level machined steel plate that has been slightly warmed and thinly coated with kerosene. Using a guide, one end of the specimen shall be placed into this

layer of molten sulphur mixture with its axis vertical Amd No. 1/2013. After a few seconds, the surplus material around the specimen may be cut away with a sharp knife and the specimen lifted off the plate. The mixture shall be allowed to harden before repeating the procedure for the other end of the specimen. The specimen shall then be checked to ensure that the capping material has adhered to the end of the specimen by lightly tapping the capping. If a hollow sound was heard, the capping compound shall be removed and the capping operation shall be repeated. Amd No. 1/2013 The cap shall not flow or fracture before the concrete fails. After testing, the capping compound shall not be re-used.

15.5.3 Tolerances

The tolerances of the prepared specimen shall be as follows:

- (a) **Flatness.** The flatness tolerance for the prepared end surfaces shall be ± 0.06 % of the core diameter, in millimetres.
- (b) **Perpendicularity.** The perpendicularity tolerance for the prepared end with respect to the axis of the specimen as datum axis shall be ± 1.0 mm.
- (c) **Parallelism.** The parallelism tolerance for the prepared top surface with respect to the bottom surface of the specimen as datum face shall be ± 2.0 mm.

15.5.4 Storage

After end preparation, all specimens shall be stored in water or a mist chamber at (27 ± 3) °C until saturated.

15.6 TESTING PROCEDURE

15.6.1 General

Specimens with cracked or loose caps shall not be tested. The specimens shall be tested within one hour of removal from the water or mist chamber, whilst they are still wet.

15.6.2 Placing the specimen in the testing machine

The bearing surfaces and the auxiliary platens of the testing machine shall be wiped clean and any water, loose grit or other material shall be removed from the ends of the specimen. The specimen shall be carefully centred on the lower platen of the machine. No packing other than auxiliary steel platens shall be used between the ends of the specimen and the platens of the testing machine.

15.6.3 Loading

The load shall be applied steadily such that the stress is increased at a rate within the range of 0.2 MPa/s to 1.0 MPa/s until no greater load can be sustained. The maximum load shall be recorded.

Normal failures are reasonably symmetrical. The appearance of the concrete and any unusual failure shall be noted.

15.6.4 Type of failure

Any unusual features in the type of failure shall be recorded. Examples of satisfactory failures and of some unsatisfactory failures as shown in Figure 17.

15.7 CALCULATION AND EXPRESSION OF RESULTS

15.7.1 Compressive strength of core

The compressive strength of the core shall be calculated by dividing the maximum load by the cross-sectional area as calculated from the average diameter. The results shall be expressed to the nearest 0.1 MPa.

NOTE. *The presence of reinforcement in a core cut from reinforced concrete may affect the result.*

15.7.2 Estimated in-situ cube strength

For cores free of reinforcement, the estimated in-situ cube strength, F_{ce} shall be calculated to the nearest 0.5 MPa from the equation:

$$F_{ce} = \frac{D}{1.5 + \frac{1}{\alpha}} \times \frac{\text{measured compressive strength of core}}{\text{(to nearest 0.1 MPa)}} \quad \text{---- (15 - 1)}$$

where

- F_{ce} is the estimated in-situ cube strength
- D is 2.5 for cores drilled horizontally (for precast units perpendicular to height when cast) or 2.3 for cores drilled vertically (for precast units parallel to height when cast)
- α is the length (after end preparation)/diameter ratio

NOTE. *It should be noted that in-situ strengths estimated from the above formula cannot be equated to standard cube strengths.*

For cores with reinforcement perpendicular to the core axis, the estimated in-situ cube strength shall be calculated by multiplying the strength of core obtained from equation (15 - 1) by the following factors:

- (a) for cores containing a single bar:

$$1.0 + 1.5 \frac{\phi_r d}{\phi_c L} \quad \text{---- (15 - 2)}$$

- (b) for specimens containing two bars no further apart at their closest point than the diameter of the larger bar, only the bar corresponding to the higher value of $\phi_r d$ need be considered. If the bars are further apart, their combined effect should be assessed by using the factor:

$$1.0 + 1.5 \frac{\sum \phi_r d}{\phi_c L} \quad \text{---- (15 - 3)}$$

where

- ϕ_r is the diameter of reinforcement
- ϕ_c is the diameter of specimen
- d is the distance of axis of bar from nearer end of specimen after end preparation
- L is the length of specimen after end preparation

15.8 TEST REPORT

The report shall affirm that the specimens were prepared and tested in accordance with this Standard.

15.8.1 Information to be provided by the producer of the test specimens for inclusion in the test report

The following information shall be provided:

- (a) Name of project and place where core was taken.
- (b) Specified concrete strength, if known.
- (c) Concrete mix details, if known.
- (d) Admixtures used, if known.
- (e) Identification number of the core.
- (f) Date of drilling.
- (g) Direction of drilling relative to the direction of casting e.g. vertically, horizontally or diagonally.
- (h) Conditions of storage.
- (i) Age of concrete at time of coring, if known.
- (j) Name and signature of person responsible for taking cores.

15.8.2 Information to be provided by the test laboratory for inclusion in the test report

The following information shall be provided:

- (a) Identification number of the specimen.
- (b) Date of receipt of the specimen.

- (c) Average diameter.
- (d) Saturated density of the specimen.
- (e) Length before and after preparation, and location in relation to the length received.
- (f) Method used for the preparation of specimen (cutting, grinding or capping).
- (g) Condition of specimen after cutting including compaction of concrete, honeycombing, distribution of materials, classification of voids and presence of cracks.
- (h) Size, position and spacing of any reinforcement.
- (i) Description of aggregate, including maximum size and particle shape.
- (j) A set of four photographs of the core as received taken at 90° intervals.
- (k) Date of test.
- (l) Age of specimen at date of test, if known.
- (m) Maximum load at failure.
- (n) Measured compressive strength and estimated in-situ cube strength.
- (o) Appearance of concrete and type of fracture (Figure 17).
- (p) Other remarks if any.
- (q) Name and signature of person responsible for the test.

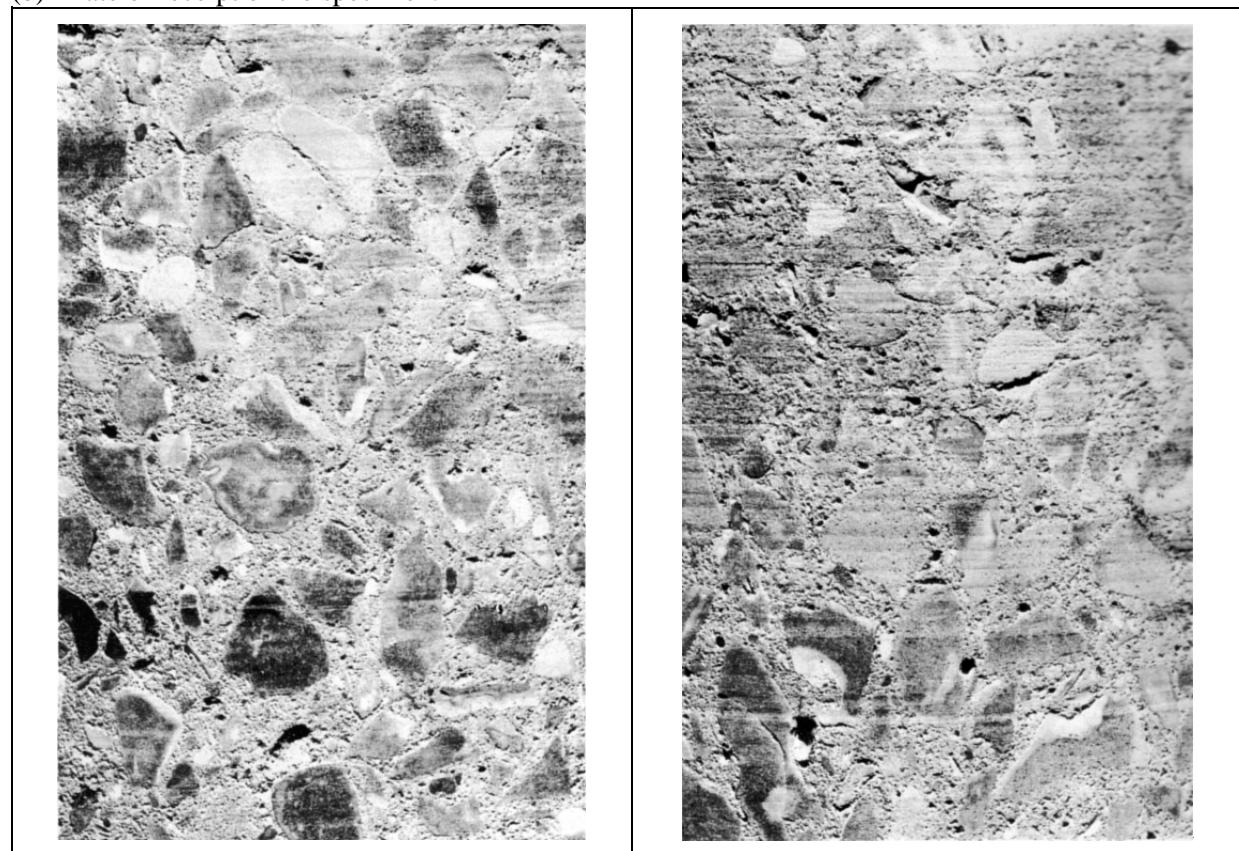


Figure 16 (a) Excess voidage = 0

Figure 16 (b) Excess voidage = 0.5 %

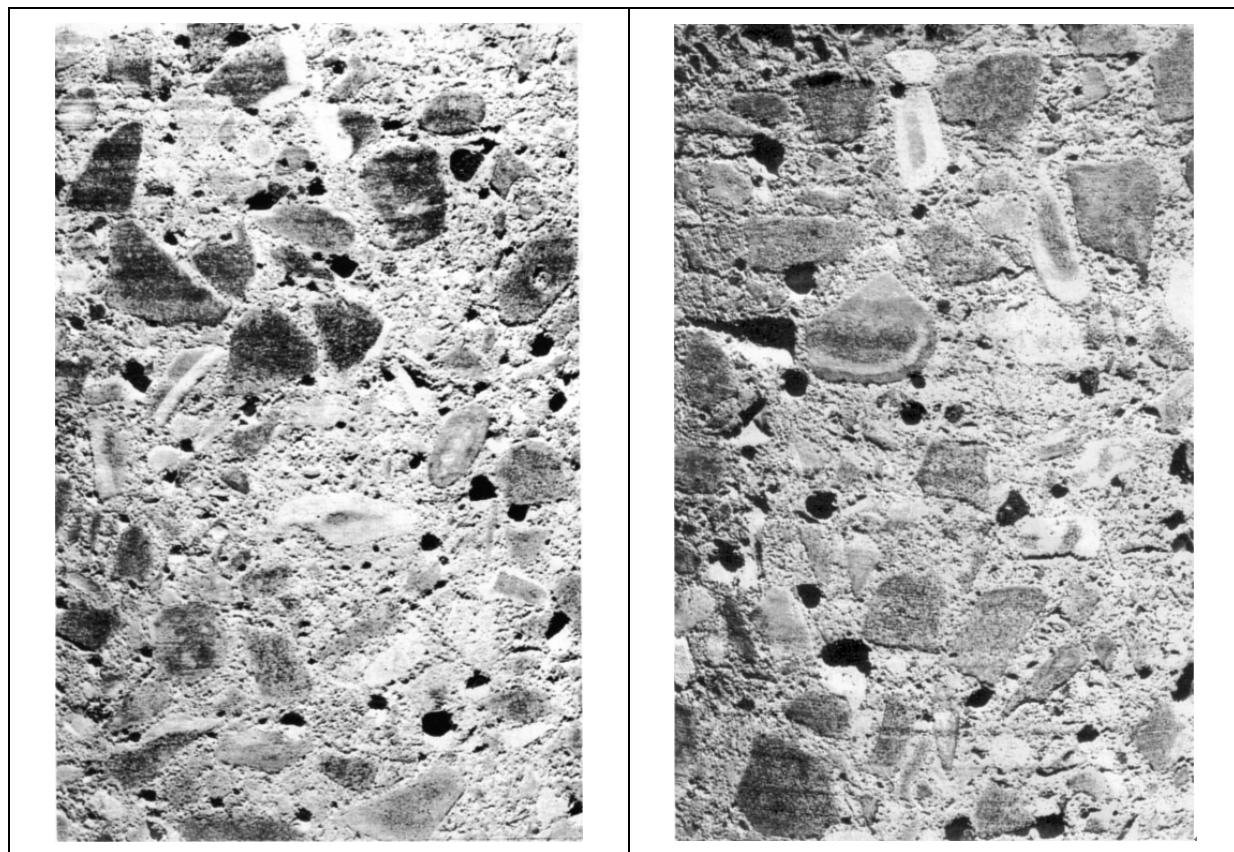


Figure 16 (c) Excess voidage = 1.5 %

Figure 16 (d) Excess voidage = 3.0 %

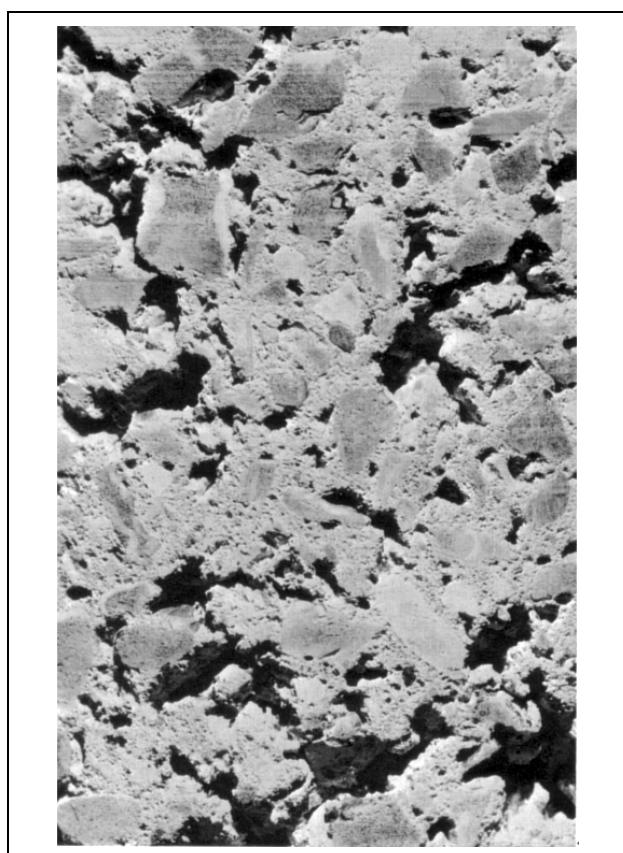
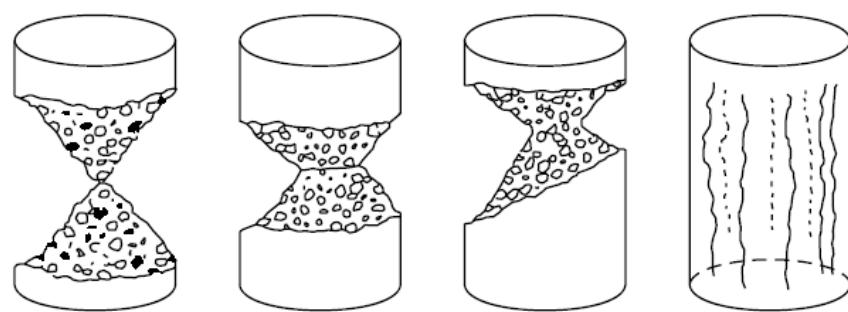
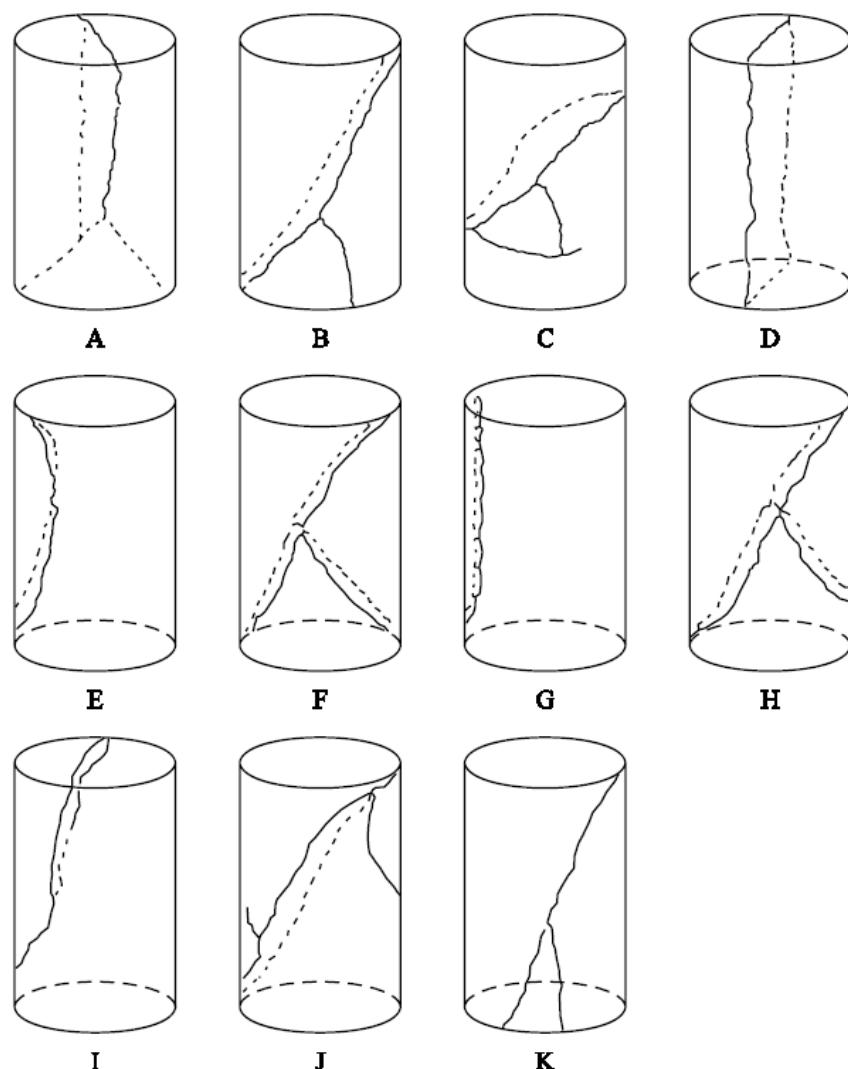


Figure 16 (e) Excess voidage = 13.0 %



Satisfactory failure of cylinder specimen



Some unsatisfactory failures of cylinder specimens

Figure 17 - Failure Patterns of Cylinders

SECTION 16

DETERMINATION OF DENSITY OF HARDENED CONCRETE

16.1 SCOPE

This Section describes the methods of determining the density of hardened concrete. The method is applicable to lightweight, normal-weight and heavy-weight concrete.

16.2 APPLICATION

The as-received density generally applies to specimens cut from a structure and tested on arrival in the laboratory or to moulded specimens which have not been cured to a saturated state.

The as-received density may be required in the following tests:

- (a) Compressive strength of cubes (Section 12).
- (b) Tensile splitting strength (Section 13).
- (c) Flexural strength (Section 14).

The saturated and oven-dried densities provide a convenient basis for comparison as the conditions are reproducible, but may not be representative of the in-situ condition.

Saturated density may be required in the following tests:

- (a) Compressive strength of cube (Section 12).
- (b) Tensile splitting strength (Section 13).
- (c) Flexural strength (Section 14).
- (d) Compressive strength of cores (Section 15).
- (e) Modulus of Elasticity (Section 17).

16.3 DEFINITIONS

Density (ρ) is the mass of a unit volume of hardened concrete expressed in kilograms per cubic metre.

$$\rho = \frac{m}{V} \quad \text{----- (16 - 1)}$$

where

m can be m_1 = mass of the as-received specimen in air (in kg)

| | |
|------------------|---|
| or m_2 | = mass of the saturated specimen in air (in kg) |
| or m_3 | = mass of the oven-dried specimen in air (in kg) |
| V can be V_1 | = the volume of the specimen calculated from its measured dimensions (in m^3) |
| V_2 | = the volume of the specimen determined by the displacement of water (in m^3) |

NOTE. Determination of the volume by water displacement is to be preferred, especially for cut or cored specimens.

16.4 APPARATUS

The following apparatus is required:

- (a) Water container (see C1. C2.2).
- (b) Caliper (see C1. C7).
- (c) Balance with stirrup (see C1. C8.2).
- (d) Oven at 105 °C (see C1. C17.1).

Further details of the apparatus are given in Appendix C.

16.5 TEST SPECIMENS

The volume of the specimen shall be not less than $40 d^3$, where d is the nominal maximum size of the aggregate. If the shape or size of the sample is such that it is not possible to use all of it, a smaller specimen may be taken from the original, provided that the prepared specimen is representative of the as-received sample.

16.6 PROCEDURE FOR DETERMINATION OF THE VOLUME BY CALCULATION

Only specimens of regular shape shall be measured. All measurements of dimensions shall be recorded to the nearest 0.1 millimetre.

16.6.1 Cylindrical specimens

The length of the specimen parallel to the axis shall be measured at four evenly distributed positions and a pair of measurements of the diameter shall be taken right angles to each other at each one-third point along the length. The average length and diameter shall be calculated and the volume V_1 of the specimen (in m^3) determined.

16.6.2 Rectilinear specimens (excluding cubes)

The length parallel to the axis of the specimen shall be measured at the centre of the four edges. The width shall be measured with a calliper at two points across each pair of opposite faces. The average width shall be calculated and the volume V_1 of the specimen (in m^3) determined.

16.6.3 Cubes

The cube dimensions between the centres of the three pairs of opposing faces shall be measured with a calliper. The volume V_1 of the cube (in m^3) shall be determined from the measured dimensions.

16.7 PROCEDURE FOR DETERMINATION OF THE VOLUME BY WATER DISPLACEMENT

NOTE. *The water displacement method is not applicable to specimens of no-fines concrete, lightweight aggregate concrete with large pores or samples where the moisture content is not to be altered. However, the application of a thin water impermeable layer can make this method practicable. Water shall be within $(25 \pm 5)^\circ\text{C}$ where density of water is 997 kg/m^3 .*

16.7.1 Procedure

If the specimen is not saturated, it shall first be immersed in water for 30 minutes. The specimen shall then be placed on the stirrup and fully immersed in water. The stirrup shall not touch the bottom of the tank and air bubbles shall not be allowed to be trapped on the surfaces of the specimen or the stirrup. The completely immersed specimen shall be weighed and its apparent mass m_w recorded after correcting for the apparent mass of the empty stirrup. The apparent mass of the empty stirrup

shall be determined by weighing it in water to the same depth as when holding the specimen.

The specimen shall be wiped to remove surface water and then weighed in air and its mass m_a recorded.

16.7.2 Calculation

Taking the density of water as 997 kg/m^3 , the volume V_2 of the specimen shall be calculated from the following equation:

$$V_2 = \frac{m_a - m_w}{997} \quad \text{----- (16 - 2)}$$

16.8 PROCEDURE FOR DETERMINATION OF AS-RECEIVED DENSITY

16.8.1 Procedure

The specimen as-received shall be weighed in air and its mass m_1 recorded. The volume of the specimen may be determined in accordance with C1. 16.6 or C1. 16.7.

16.8.2 Calculation

The as-received density shall be calculated using the appropriate equation:

- (a) for volume calculated in accordance with C1. 16.6

$$\rho_1 = \frac{m_1}{V_1} \quad \text{----- (16 - 3)}$$

- (b) for volume obtained by water displacement in accordance with C1. 16.7

$$\rho_2 = \frac{m_1}{V_2} \quad \text{----- (16 - 4)}$$

16.9 PROCEDURE FOR DETERMINATION OF SATURATED DENSITY

16.9.1 Procedure

The specimen shall be fully immersed in water at $(27 \pm 3)^\circ\text{C}$ until constant mass is achieved. This state shall be regarded as having been

reached when two measurements taken 24 hours apart give a difference of less than 0.2 % in the mass of the wet specimen in air. Before each weighing, surplus water from the surface shall be removed with a moist cloth. The constant mass m_2 shall be recorded. Specimens cured in water in accordance with Section 10 of this Standard for more than three days immediately prior to testing may be assumed to be saturated to a constant mass for this test. The volume of the specimen shall be determined in accordance with C1. 16.6 or 16.7.

16.9.2 Calculation

The saturated density shall be calculated using the appropriate equation:

- (a) for volume calculated in accordance with C1. 16.6

$$\rho_3 = \frac{m_2}{V_1} \quad \text{----- (16 - 5)}$$

- (b) for volume obtained by water displacement in accordance with C1. 16.7

$$\rho_4 = \frac{m_2}{V_2} \quad \text{----- (16 - 6)}$$

16.10 PROCEDURE FOR DETERMINATION OF OVEN-DRIED DENSITY

16.10.1 Procedure

The specimen shall be placed in a ventilated oven and dried at $(105 \pm 5)^\circ\text{C}$ until constant mass is achieved. This state shall be regarded as having been reached when two measurements taken 24 hours apart give a difference of less than 0.2 % in the mass of the dry specimen. Before each weighing, the specimen shall be cooled to near room temperature in a dry airtight vessel or desiccator. The constant mass m_3 shall be recorded. The volume of the specimen shall be determined in accordance with C1. 16.6 or 16.7.

16.10.2 Calculation

The oven-dried density shall be calculated using the appropriate equation:

- (a) For volume calculated in accordance with C1. 16.6

$$\rho_5 = \frac{m_3}{V_1} \quad \text{----- (16 - 7)}$$

- (b) for volume obtained by water displacement in accordance with C1. 16.7

$$\rho_6 = \frac{m_3}{V_2} \quad \text{----- (16 - 8)}$$

16.11 CALCULATION AND EXPRESSION OF RESULTS

The density of each specimen shall be calculated using the appropriate equation given in C1. 16.7, 16.8, 16.9 or 16.10 and shall be expressed to the nearest to 10 kg/m^3 .

16.12 TEST REPORT

The report shall affirm that the test was carried out in accordance with this Standard and shall include the following:

- (a) Identification number of the specimen.
- (b) Date of receipt of the specimen at the laboratory.
- (c) Condition of the specimen when received (including poor compaction, honeycombing and incorrect dimensions).
- (d) Date of test.
- (e) Method of determination of volume.
- (f) Measured dimensions and shape of the specimen.
- (g) Mass of specimen (as-received, saturated or oven-dried).
- (h) Type of density measured, (i.e. as-received, saturated or oven-dried) and values obtained.
- (i) Name and signature of person responsible for the test.

SECTION 17

DETERMINATION OF STATIC MODULUS OF ELASTICITY IN COMPRESSION

17.1 SCOPE

This Section describes the method of determining the static modulus of elasticity in compression of hardened concrete, on test specimens which may be cast or taken from a structure.

17.2 DEFINITION

Static modulus of elasticity in compression, E_c , is the ratio between compressive stress and strain, expressed in terms of the secant modulus. The secant modulus in MPa is calculated from the equation:

$$E_c = \frac{\Delta_\sigma}{\Delta_\varepsilon} \quad \text{----- (17 - 1)}$$

Where Δ_σ and Δ_ε are the differences in stress and strain respectively, between a basic loading level of 0.5 MPa and an upper loading level of one-third of the compressive strength of the concrete.

17.3 APPARATUS

The following apparatus is required:

- (a) Compression testing machine (see C1. C5).
- (b) Strain measuring apparatus (see C1. C18).

Further details of the apparatus are given in Appendix C.

17.4 TEST SPECIMENS

17.4.1 Moulded specimens

Moulded specimens shall be 150 mm diameter by 300 mm long cylinders, made, cured and stored in accordance with Sections 9 and 10 of this Standard. Two standard 150 mm cubes shall also be made from concrete of the same batch and cured under same condition as the specimen. If the upper surface of the cylinder has not been prepared whilst it is still workable, it shall be allowed to harden and the upper surface

prepared by grinding or capping. After grinding or capping, the specimen shall be stored in water or a mist chamber until saturated.

17.4.1.1 Grinding of hardened cylinder

After the concrete has hardened, the upper surface shall be ground until it complies with the tolerances stated in C1. 17.4.1.3. Only water shall be used as a coolant during grinding.

17.4.1.2 Sulphur capping of hardened cylinder

Caps shall be made as thin as possible and shall not exceed 10 mm thickness at any point.

The capping material shall consist of a mixture of equal parts by weight of sulphur and fine siliceous sand (most of which passes a 300 µm woven wire sieve and is retained on a 150 µm woven wire sieve) together with a small proportion (1 % to 2 %) of carbon black or a small proportion (2 % to 4 %) of polysulphide rubber. The mixture shall be heated to a temperature of 130 °C to 150 °C and then allowed to cool slightly while being stirred continuously. The mixture shall be poured onto a level machined steel plate that has been slightly warmed and thinly coated with kerosene. Using a guide, the specimen shall be placed into this layer with its axis vertical. After a few seconds, the surplus material around the specimen may be cut away with a sharp knife and the specimen lifted off the plate. The cap shall not flow or fracture before the concrete fails.

17.4.1.3 Tolerances

If the upper end is ground or capped when hardened, the end shall comply with the following limits:

- (a) **Flatness.** The flatness tolerance for the prepared surface shall be ± 0.06 mm.
- (b) **Parallelism.** The parallelism tolerance for

the prepared surface with respect to the lower surface of the cylinder as datum face shall be ± 2.0 mm.

17.4.2 Cores

Specimens drilled out of a structure shall be drilled, stored and capped in accordance with Section 15 of this Standard. The minimum dimension of the core shall be not less than three times the nominal maximum size of aggregates in the concrete nor less than 100 mm. An extra core shall be taken for the determination of the concrete strength.

17.4.3 Measurement of mass and density

The mass and saturated density of each specimen shall be determined in accordance with Section 16 of this Standard.

17.5 PROCEDURE

17.5.1 Determination of compressive strength

All specimens shall be tested within one hour of removal from the water or mist chamber, whilst they are still wet.

Just before commencing the static modulus of elasticity test, the compressive strength of the moulded specimen shall be determined from the two standard 150 mm cubes of the same batch, made and cured under similar conditions as the specimen. The cubes shall be crushed and the concrete strength converted to an equivalent cylinder strength by multiplying the cube strength by a factor of 0.8.

For concrete cores, the compressive strength shall be determined in accordance with the procedure given in Section 15 of this Standard.

The mean value of the compressive strength (i.e. equivalent cylinder strength), f_c , determines the stress to be applied in the determination of static modulus of elasticity.

17.5.2 Measuring instruments or fixing points

Measuring instruments or fixing points shall be

attached to the test specimen axially in such a way that the gauge points are equidistant from the two ends of the specimen and at a distance not less than one-quarter of the length of the test specimen ($L/4$) from its ends. At least one pair of measurements shall be taken on opposite sides of the specimen.

NOTE. *Where fixing points for extensometer are required, threaded inserts cast into the specimen are preferred.*

If adhesives are used for the fixing points they should be rapid setting and set hard. The specimen shall be removed from the curing tank or the mist chamber for as short a time as possible to allow the surface to be dried for the application of adhesive. Specimens shall not be less than seven days old when removed for this purpose. Specimens shall be placed back in the water or mist chamber for a minimum of two days before testing.

17.5.3 Loading

The test specimen, with the measuring instruments or fixing points attached axially, shall be placed centrally in the machine. The basic stress of 0.5 MPa (σ_b) shall be applied, and the strain gauge readings at each measurement line shall be recorded.

The load shall be applied steadily and without shock such that the stress is increased at a constant rate within the range 0.5 MPa/s to 0.7 MPa/s until the stress is equal to one-third of the compressive strength of the concrete ($\sigma_a = f_c/3$).

NOTE. *The preferred rate is 0.6 MPa/s.*

The stress shall be maintained for 60 seconds and the strain readings at each measurement line during the succeeding 30 seconds shall be recorded. If the individual strains are not within a range of ± 10 % of their mean value at σ_a , the test specimen shall be recentered and the test repeated. If it is not possible to reduce the differences to within this range, the test shall be stopped.

When the centring is sufficiently accurate, the load shall be reduced to the level of the basic stress at the same rate as during loading. At least two additional loading cycles, using the same loading and unloading rate, shall be carried out and the stresses (σ_a and σ_b) maintained

constant for a period of 60 seconds. After completion of the last loading cycle and a waiting period of 60 seconds under the stress $\sigma_b = 0.5$ MPa, the strain reading at the various measurement lines ε_b , during the succeeding 30 seconds shall be recorded.

The specimen shall be reloaded to stress σ_a at the specified rate, and the strain reading ε_a at the various measurement lines taken within 30 seconds shall be recorded.

When all elasticity measurements have been completed, the load on the test specimen shall be increased, at the specified rate, until failure of the specimen occurs. If the compressive strength of the specimen differs from f_c by more than 20 %, this shall be noted in the test report.

17.6 CALCULATION AND EXPRESSION OF RESULTS

The mean strain ε_a and ε_b respectively shall be calculated.

The static modulus of elasticity in compression E_c (in MPa) is given by the equation:

$$\frac{\Delta\sigma}{\Delta\varepsilon} = \frac{\sigma_a - \sigma_b}{\varepsilon_a - \varepsilon_b} \quad \text{----- (17 - 2)}$$

Where

σ_a is the upper loading stress (in MPa)
($\sigma_a = f_c/3$)

- σ_b is the basic stress (0.5 MPa)
- ε_a is the mean strain under the upper loading stress
- ε_b is the mean strain under the basic stress

The result shall be expressed to the nearest 500 MPa for values over 10 000 MPa, and to the nearest 100 MPa for values below 10 000 MPa.

17.7 TEST REPORT

The report shall affirm that the tests were carried out in accordance with this Standard and shall include the following:

- (a) Identification number of the specimen.
- (b) Date of receipt of the specimen.
- (c) Condition of the specimen when received, and any surface treatment.
- (d) Curing and storage report.
- (e) Date of test.
- (f) Saturated density.
- (g) Concrete cube strength or core strength at date of testing.
- (h) Maximum applied stress during test ($f_c/3$).
- (i) Compressive strength of test specimen.
- (j) Static modulus of elasticity.
- (k) Appearance of the concrete and type of fracture (Figure 17).
- (l) Name and signature of person responsible for the test.

NOTE. The report on the production and curing of the test specimen may conveniently be included as part of the testing report.

SECTION 18

DETERMINATION OF DEPTH OF PENETRATION OF WATER UNDER PRESSURE

18.1 SCOPE

This Section describes the method of determining the depth of penetration of water under pressure in hardened concrete which has been water cured.

The test specimen shall be cubes or cylinders of length of edge, or diameter, not less than 150 mm.

18.2 APPARATUS

The following apparatus is required:

Water penetration test apparatus
(Figure 18) (see C1. C19).

Further details of the apparatus are given in Appendix C.

NOTE. *Tap water may be used.*

18.3.3 Examination of specimen

After the pressure has been applied for the specified time, the specimen shall be removed from the apparatus. The face on which the water pressure was applied shall be wiped to remove excess of water. The specimen shall be split in half, perpendicularly to the face on which the water pressure was applied. When splitting the specimen, and during the examination, the face of the specimen exposed to the water pressure shall be placed on the bottom. As soon as the split face has dried to such an extent that the water penetration front can be clearly seen, the water front on the specimen shall be marked. The maximum depth of penetration under the test area shall be measured and recorded to the nearest millimetre.

18.3 PROCEDURE

18.3.1 Preparation of specimen

Immediately after the test specimen is de-moulded, the surface of the specimen to be exposed to water pressure shall be roughened with a wire brush.

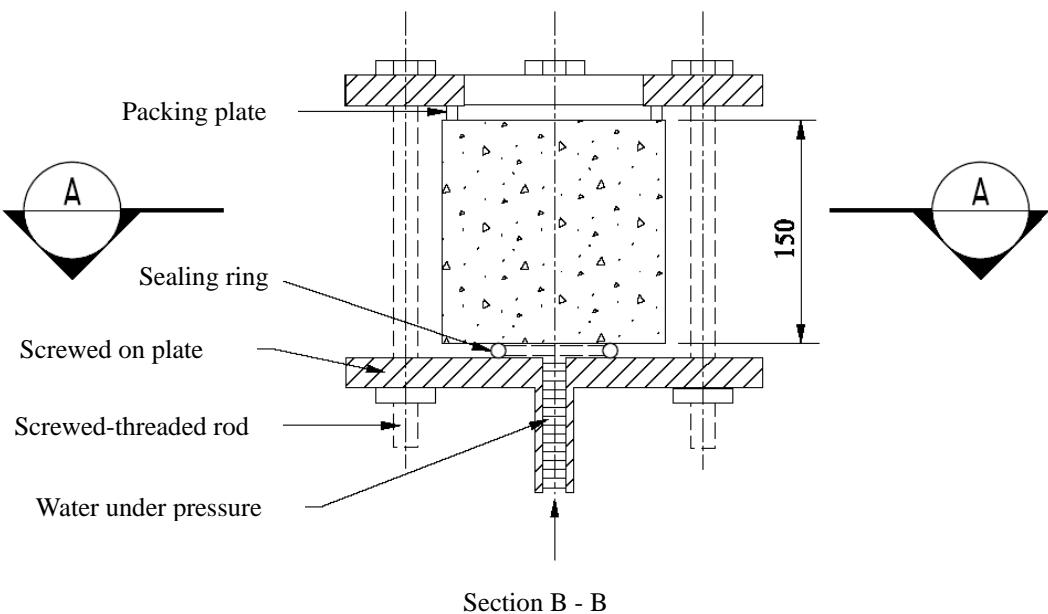
18.3.2 Application of water pressure

The specimen shall be at least 28 days old. Water pressure shall not be applied to a trowelled surface of the specimen. The specimen shall be placed in the apparatus and a water pressure of (500 ± 50) kPa shall be applied for (72 ± 2) hrs. During the test, the appearance of the surfaces of the test specimen not exposed to the water pressure shall be observed periodically to note the presence of water. If leakage is observed, the validity of the result shall be considered and the fact shall be recorded.

18.4 REPORT

The report shall affirm that the tests were carried out in accordance with this Standard and shall include the following:

- (a) Identification of the test specimen.
- (b) Date at which the test was started.
- (c) Description of the specimen.
- (d) Direction of application of water pressure with respect to the casting direction.
- (e) Maximum depth of penetration, in millimetres.
- (f) Any leakage and consideration of the validity of the result (if appropriate).
- (g) Any deviation from standard test method.
- (h) A declaration by the person technically responsible for the test that it was carried out in accordance with this Standard, except as noted in item (g).
- (i) Name and signature of person responsible for the test.



NOTE:

1. The apparatus shall allow the other surface of the test specimen to be observed.
2. The water pressure may be applied to the surface of the test specimen either from the bottom, or the top.
3. A necessary seal shall be made of rubber or other similar material.
4. The dimensions of test area shall be approximately half of the length of the edge or diameter of the test surface.
5. All dimensions are in millimetres.

Figure 18 - Apparatus for Determination of Depth of Water Penetration

SECTION 19

DETERMINATION OF CONCRETE'S ABILITY TO RESIST CHLORIDE ION PENETRATION

19.1 SCOPE

This method sets out the procedure for determination of electrical conductance of concrete to provide a rapid indication of its resistance to penetration of chloride ions.

19.2 APPARATUS

The following apparatus is required :

- (a) Vacuum saturation apparatus (see Cl. C20).
- (b) Specimen cell sealant (see Cl. C21).
- (c) Voltage cell and data readout apparatus (Figure 19 & Figure 20) (see Cl. C22).
- (d) Coating apparatus and materials.

Further details of the apparatus are given in Appendix C.

19.3 REAGENTS

- (a) Sodium chloride (NaCl) solution shall be 3.0 % by mass (reagent grade) in distilled water.
- (b) Sodium hydroxide (NaOH) solution shall be 0.3 N (reagent grade) in distilled water.

NOTE. Before using NaOH, review: (1) the safety precautions for using NaOH; (2) first aid for burns; and (3) the emergency response to spills, as described in the manufacturer's Material Safety Data Sheet or other reliable safety literature. NaOH can cause very severe burns and injury to unprotected skin and eyes. Suitable personal protective equipment should always be used. These should include full-face shields, rubber aprons, and gloves impervious to NaOH. Gloves should be checked periodically for pinholes.

19.4 PREPARATION OF TEST SPECIMENS

19.4.1 Source of sample

Sample preparation and selection shall be depended on the purpose of the test. For evaluation of materials or their proportions, samples may be (a) cores from test slabs or from large diameter cylinders or (b) 100 mm diameter cast cylinders. For evaluation of structures,

samples may be (a) cores from the structure or (b) 100 mm diameter cylinders cast and cured at the field site. When cylinders are cast in the field to evaluate a structure, care must be taken that the cylinders receive the same treatment as the structure, for example, similar degree of consolidation, curing, and temperature history during curing.

NOTE. The maximum allowable aggregate size has not been established for this test. Users have indicated that test repeatability is satisfactory on specimens from the same concrete batch for aggregates up to 20.0 mm nominal maximum size.

The cores or field-cured cylinders shall be transported to the laboratory in sealed (tied) plastic bags.

19.4.2 Size of specimen

Slice (50 ± 3) mm shall be cut from core or cylinder obtained from clause 19.4.1 using the water-cooled diamond saw or silicon carbide saw from the top of the core or cylinder, with the cut parallel to the top of the core. This slice will be the test specimen. Any burrs on the end of the specimen shall be removed using a belt sander.

Special processing is necessary for core samples where the surface has been modified, for example, by texturing or by applying curing compounds, sealers, or other surface treatments, and where the intent of the test is not to include the effect of the modifications. In those cases, the modified portion of the core shall be removed and the adjacent (50 ± 3) mm slice shall be used for the test.

NOTE. The test method shall be applied to 100 mm nominal diameter specimens. This includes specimens with actual diameters ranging from 95 mm to 102 mm. Other specimen diameters may be tested with appropriate changes in the applied voltage cell design. For specimen diameters other than 95 mm, the test result value for total charge passed must be adjusted following the procedure in clause 19.7.3. For specimens with diameters less than 95 mm, particular care must be taken in coating and mounting the specimens to ensure that the conductive solutions are able to contact the entire end areas during the test.

19.5 CONDITIONING OF SPECIMENS

19.5.1 Preparation of de-aerated water

A litre or more of tap water in a large sealable container shall be vigorously boiled. The container shall be removed from heat, capped tightly, and water shall be cooled to ambient temperature.

19.5.2 Coating of specimens

Coating shall be rapid setting, electrically nonconductive, capable of sealing side surface of concrete cores.

Slice of specimen prepared in clause 19.4.2 shall be surface dried in air for at least 1 hour. About 10 g of rapid setting coating shall be prepared and brushed onto the side surface of specimen. The specimen shall be placed on a suitable support while coating to ensure complete coating of sides. The coated specimen shall be cured according to the manufacturer's instructions or until it is no longer sticky to the touch. Any apparent holes in the coating shall be filled and additional curing time shall be allowed, as necessary.

19.5.3 De-aeration of specimens

The specimen shall be placed in beaker or other container. The container shall then be placed in vacuum desiccator. Alternatively, the specimen can be placed directly in vacuum desiccator. Both end faces of specimen must be exposed.

The desiccator shall be sealed and vacuum pump or aspirator shall be started to decrease the pressure to less than 50 mm Hg (6650 Pa) within a few minutes and the vacuum shall be maintained for 3 hour.

19.5.4 Saturation of specimens

The separatory funnel or other container (see clause 19.2 (a)) shall be filled with de-aerated water prepared in clause 19.5.1. With vacuum pump still running, the stopcock shall be opened to allow sufficient water to be drained into beaker or container to cover specimen (air shall not be allowed to enter desiccator through this stopcock). The stopcock shall then be closed

and vacuum pump shall be run for one additional hour.

19.5.5 Soaking of specimens

The vacuum line stopcock shall be closed and then the pump shall be turned off (change pump oil if a water trap is not being used). The vacuum line stopcock shall be opened to allow air to re-enter desiccator.

The specimen shall be soaked under water in the beaker for (18 ± 2) h.

19.6 PROCEDURE

19.6.1 Storage of specimens

The specimen shall be removed from water, blotted off excess water, and transferred to a sealed container which the specimen will be maintained in 95 percent or higher relative humidity.

19.6.2 Mounting of specimens

The specimen shall be mounted (all sealants other than rubber gaskets) by setting the specimen onto 850 μm brass screen and the sealant shall be applied around specimen-cell boundary. The exposed face of specimen shall be covered with an impermeable material such as rubber or plastic sheeting. The rubber stopper shall be placed in cell filling hole to restrict moisture movement. The sealant shall be cured as per manufacturer's instructions. The specimen mounting steps shall be repeated on the second half of cell.

Alternatively, the specimen shall be mounted (rubber gasket) by placing a 100 mm outside diameter by 75 mm inside diameter by 6 mm circular vulcanized rubber gasket in each half of the test cell. Then the specimen shall be inserted and the two halves of the test cell shall be clamped together to seal.

19.6.3 Filling of reagent into cells

The side of the cell containing the top surface of the specimen shall be filled with 3.0 percent NaCl solution. (That side of the cell will be

connected to the negative terminal of the power supply in clause 19.6.4). The other side of the cell (which will be connected to the positive terminal of the power supply) shall be filled with 0.3 N NaOH solution.

19.6.4 Electrical connection

The lead wires shall be attached to the cell banana posts and electrical connections shall be made to voltage application and data readout apparatus as appropriate; for example, for system listed in clause 19.2(c), and connection as shown in Figure 20.

19.6.5 Starting test

The power supply shall be turned on, set to (60.0 ± 0.1) V, and initial current reading shall be recorded. Temperatures of the specimen, applied voltage cell, and solutions shall be 20 °C to 25 °C at the time when the test is initiated, that is, when the power supply is turned on. During the test, the air temperature around the specimen shall be maintained in the range of 20 °C to 25 °C.

19.6.6 Measurement

The current shall be read and recorded at every 30 minutes up to 6 hours. If a voltmeter is being used in combination with a shunt resistor for the current reading (see Figure. 20), appropriate scale factors shall be used to convert voltage reading to amperes. Each half of the test cell must be remain filled with the appropriate solution for the entire period of the test. The test shall be terminated after 6 hour, except as discussed in the note below.

NOTE. During the test, the temperature of the solutions should not be allowed to exceed 90 °C in order to avoid damage to the cell and to avoid boiling off the solutions. Although it is not a requirement of the method, the temperature of the solutions can be monitored with thermocouples installed through the 3 mm venthole in the top of the cell. High temperatures occur only for highly penetrable concretes. If a test of a 50 mm thick specimen is terminated because of high temperatures, this should be noted in the report, along with the time of termination, and the concrete rated as having very high chloride ion penetrability.

19.7 CALCULATION AND INTERPRETATION OF RESULTS

19.7.1 Plotting of graph

A graph shall be plotted with current (in amperes) versus time (in seconds). A smooth curve shall be drawn through the data.

19.7.2 Calculation

The area underneath the curve plotted in clause 19.7.1 shall be integrated in order to obtain the ampere-seconds, or coulombs, of charge passed during the 6-h test period.

The following formula, based on the trapezoidal rule, can be used to obtain the ampere-seconds, or coulombs, of charge passed during the 6-hour test period. Alternatively, if automatic data processing equipment is used to perform the integration during or after the test and to display the coulomb value, the total charge passed is a measure of the electrical conductance of the concrete during the period of the test.

$$Q = 900 (I_0 + 2I_{30} + 2I_{60} \dots + 2I_{300} + 2I_{330} + I_{360}) \quad \text{----- (19 - 1)}$$

where

- Q is the charge passed (coulombs)
- I_0 is the current (amperes) immediately after voltage is applied
- I_t is the current (amperes) at t min after voltage is applied

19.7.3 Correction to non-standard specimen

If the specimen diameter is other than 95 mm, the value for total charge passed established in clause 19.7.2 must be adjusted. The adjustment shall be made by multiplying the value established in clause 19.7.2 by the ratio of the cross-sectional areas of the standard and the actual specimens. That is :

$$Q_s = Q_x (95/x)^2 \quad \text{----- (19 - 2)}$$

where

- Q_s is the charge passed (coulombs) through a 95 (mm) diameter specimen

Q_x is the charge passed (coulombs) through x (mm) diameter specimen
 x is the diameter (mm) of the non-standard specimen

19.7.4 Interpretation of results

Chloride Ion Penetrability can be evaluated from the results established from clause 19.7.2 by using Table 5 below :

Table 5 - Chloride Ion Penetrability Based on Charge Passed

| Charge Passed (Coulombs) | Chloride Ion Penetrability |
|--------------------------|----------------------------|
| >4000 | High |
| 2,000-4,000 | Moderate |
| 1,000-2,000 | Low |
| 100-1,000 | Very Low |
| <100 | Negligible |

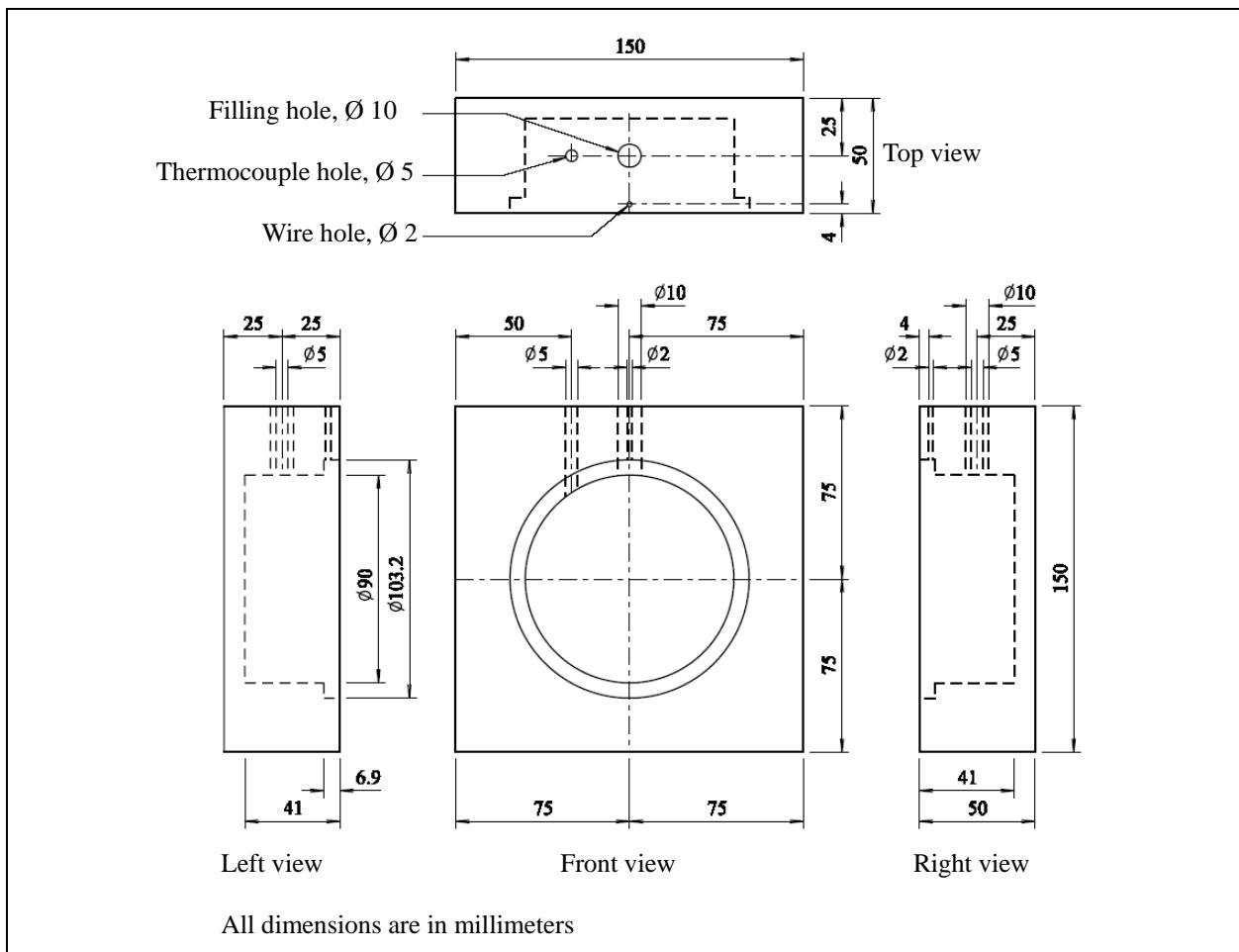
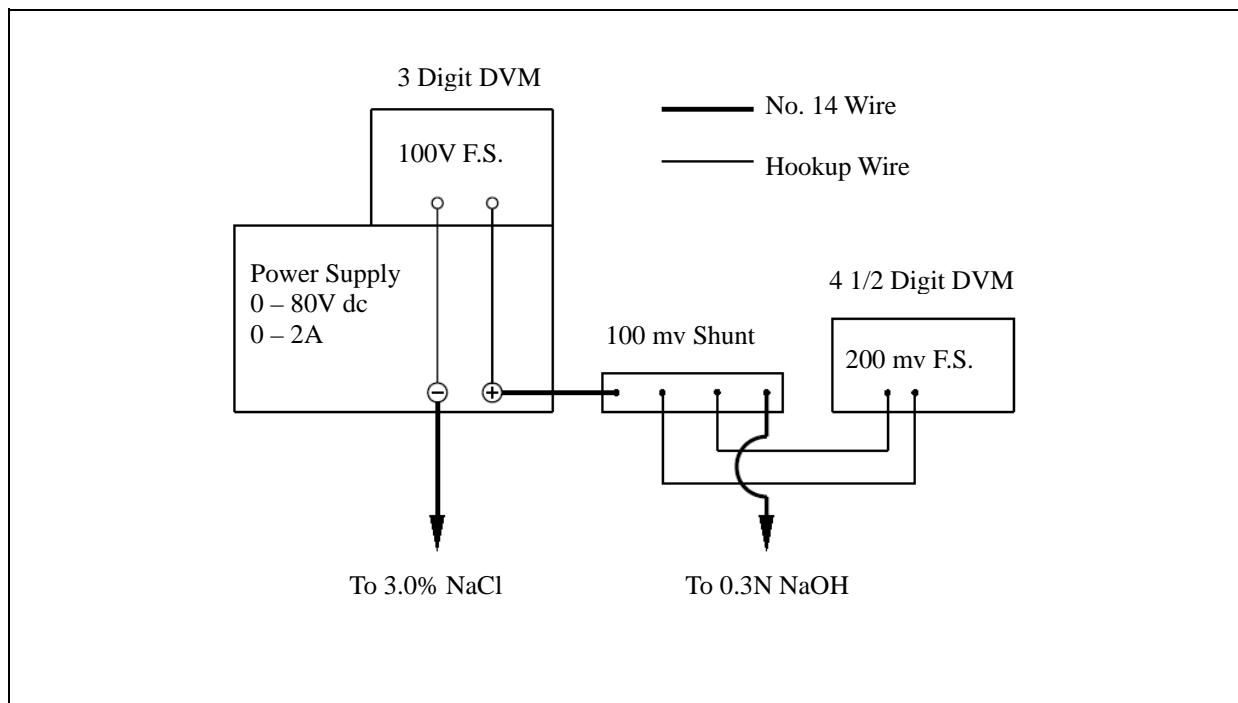
NOTE. Factors which are known to affect chloride ion penetration include : water-cement ratio, the presence of polymeric admixtures, sample age, air-void system, aggregate type, degree of consolidation, and type of curing.

19.8 TEST REPORT

The report shall affirm that the test was carried

out in accordance with this Standard and shall include the following :

- (a) Source of core or cylinder, in terms of the particular location the core or cylinder represents.
- (b) Identification number of the core or cylinder specimen.
- (c) Location of specimen within core or cylinder.
- (d) Type of concrete, including binder type, water cement ratio, concrete grade and other relevant data supplied with samples.
- (e) Description of specimen, including presence and location of reinforcing steel, presence and thickness of overlay, and presence and thickness of surface treatment.
- (f) Curing history of specimen.
- (g) Unusual specimen preparation, for example, removal of surface treatment.
- (h) Date and age of test.
- (i) Diameter and length of specimen to the nearest mm.
- (j) Duration of test to the nearest min.
- (k) Total electric charge passed over the test period to the nearest coulomb.
- (l) Quantitative chloride ion penetrability equivalent to the calculated electric charge passed (from Table 5).
- (m) Name and signature of person responsible for the test.

**Figure 19 - Applied Voltage Cell****Figure 20 – Example of electrical Block Diagram**

SECTION 20

DETERMINATION OF COMPRESSIVE STRENGTH OF CONCRETE CYLINDERS

20.1 SCOPE

This Section describes the method of determining the compressive strength of concrete cylinders.

20.2 APPARATUS

The following apparatus is required:

- (a) Compression testing machine (see Cl. C5).
- (b) Grinding equipment for concrete cylinders (see Cl. C15.1).
- (c) Calliper (see Cl. C7).
- (d) Weighing equipment of 20 kg Capacity (see Cl. C8.1).
- (e) Feeler gauges (see Cl. C9).
- (f) Steel plate (see Cl. C16).

Further details of the apparatus are given in Appendix C.

20.3 TEST SPECIMENS

20.3.1 Inspection of specimen

All cylinders shall be visually inspected. Any cylinder which was unprotected on delivery or has unsatisfactory features shall be recorded as such. A cylinder having a broken edge of length 20 mm or more shall be recorded as ‘a cylinder with damaged edge’.

20.3.2 Specimen size

Moulded specimen shall be a 150 mm diameter by 300 mm long cylinder made, cured and stored in accordance with Sections 9 and 10 of this Standard.

20.3.3 Mass and density

Each specimen shall be weighed to the nearest 10g and the as-received or saturated density determined in accordance with Section 16.

20.4 PREPARATION OF SPECIMENS

20.4.1 Preparation of end faces

The end faces of the cylinder shall be ground until they comply with the tolerances in Cl. 20.4.3. For cylinders with anticipated strength up to approximately 50 MPa, the capping method may be used as an alternative.

20.4.2 Capping

Capping shall be in accordance with Cl. 15.5.2 of this Standard, except that the caps shall not exceed 5 mm thick.

20.4.3 Tolerances

After the end faces are ground or capped, they shall comply with the following requirements:

- (a) **Dimensions.** The dimensional tolerances of the cylinder shall be ± 15 mm on its length and ± 1 mm on its diameter.
- (b) **Flatness.** The flatness of the end faces shall be ± 0.09 mm.
- (c) **Perpendicularity.** The perpendicularity of the side with respect to the end faces shall be ± 0.5 mm.

20.4.4 Storage

After the preparation of end faces, the specimen shall be stored in water or a mist chamber at (27 ± 3) °C until saturated.

20.5 PROCEDURE

20.5.1 General

Specimens with cracked or loose caps shall not be tested. The specimens shall be tested within one hour of removal from the water or mist chamber, whilst they are still wet. Surface water and grit on the specimen shall be wiped off and projecting fins removed.

20.5.2 Placing the specimen in the testing machine

The load-bearing faces and the auxiliary platens of the testing machine shall be wiped clean and any water, loose grit or other material shall be removed from the ends of the specimen. The specimen shall be carefully centred on the lower platen of the machine. No packing other than auxiliary steel platens shall be used between the ends of the specimen and the platens of the testing machine.

20.5.3 Loading

The load shall be applied steadily and without shock such that the stress is increased at a constant rate within the range (0.6 ± 0.2) MPa/s until no greater load can be sustained. The maximum load applied to the specimen shall be recorded.

20.5.4 Type of failure

Any unsatisfactory type of failure shall be recorded. Examples of satisfactory failures and of some unsatisfactory failures are shown in Figure 17.

20.6 CALCULATION AND EXPRESSION OF RESULTS

The compressive strength of the cylinder shall be calculated by dividing the maximum load by the cross-sectional area of the cylinder, which shall be calculated from the measured dimensions. The result shall be expressed to

the nearest 0.1 MPa.

20.7 TEST REPORT

The report shall affirm that the test was carried out in accordance with this Standard and shall include the following:

- (a) Identification number of the specimen.
- (b) Date of receipt of the specimen at the laboratory.
- (c) Date of test.
- (d) Age of the specimen at test.
- (e) Condition of the specimen when received (including poor compaction, honeycombing, incorrect dimensions or out of perpendicularity).
- (f) Curing report, if cured in laboratory.
- (g) Measured dimensions of specimen.
- (h) Type of preparation for the end faces of the specimen, by grinding/capping.
- (i) Mass of the specimen (as-received or saturated).
- (j) Density of the specimen (as-received or saturated, and the method of determining the volume).
- (k) Maximum load at failure.
- (l) Compressive strength of cylinder.
- (m) The closest type of unsatisfactory failure, where appropriate.
- (n) Other remarks if any.
- (o) Name and signature of person responsible for the test.

NOTE. The reports on the production and curing of the test specimen (see Sections 9 and 10) may conveniently be added to this report. The density of the specimen shall be calculated using the measured dimensions or the volume obtained from the water displacement method. Amd No. 1/2013

SECTION 21

CHEMICAL ANALYSIS OF HARDENED CONCRETE

21.1 SCOPE

This Section comprises the sampling procedures, sample treatment, plus testing methods for determining the cement content, aggregate content, original water content, type of cement, type of aggregate, chloride content, sulphate content and carbonate content on a sample of hardened concrete.

The test scope here shall only applies to concrete made with Portland cement. For concrete made with non-Portland cement, plus the testing of PFA content, shall fall outside the test scope under this Section 21.

21.2 GENERAL

21.2.1 Reagents

All reagents shall be of analytical reagent quality, and water shall be distilled or deionised.

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

Solutions of solid reagents shall be filtered if the solution is not clear.

Proprietary reagents of equal quality to those described in the Standard may be used as alternatives.

21.2.2 Apparatus

A laboratory shall be fully equipped with common apparatus such as digital balances, beakers, volumetric flasks, pipettes, burettes, filtering units, magnetic stirrer, etc. Only those apparatus, that are vital for conducting the testing of hardened concrete shall be stated under this Section.

Any volumetric glassware used shall meet at least Class B accuracy according to relevant British Standard(s). Digital balances shall meet an accuracy of at least 0.5 mg.

The filter papers used in gravimetric analysis using ignition shall be ashless.

21.3 SAMPLING

21.3.1 General

When doubt(s) appears concerning the quality of the concrete, it is a normative practice to conduct the relevant test(s) under this Section. For instance, doubts can arise from the quality of a mass of concrete, and hence the subsequent sampling techniques. In this connection, a mutual agreement between a customer and a testing organization, on details of the concrete sampling technique(s) and sampling procedures fit for conducting concrete testing work, should be recorded clearly by the testing organization.

21.3.2 Section of Sample

When selecting the concrete sample, the following requirements shall be observed:

- (a) No linear dimension of the sample shall be less than five times the nominal maximum size of the largest aggregate.
- (b) If original water content is to be determined, the sample shall be in a single piece with no cracks visible to the naked eye.
- (c) The minimum mass of the sample 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.
- (d) All foreign matter and reinforcement shall be avoided unless they are subject to test.

The sample shall be clearly and indelibly labelled with full particulars, including the date, exact position from which the sample was taken, the method of sampling and any other relevant information. The sample shall be enclosed in a heavy-duty polyethylene bag and sealed or tied securely.

21.3.3 Number of samples

A minimum of two (i.e. four recommended) representative samples, that shall be taken from different locations, and shall be tested separately, shall be applicable to the testing of the composition of concrete, having a volume up to 10 m³. When a large batch of concrete units are to be tested, at least ten representative samples shall be taken from different locations, and followed by separate analyses.

21.3.4 Additional material

Information on the composition of the cement and aggregate used in the concrete will enable more accurate results to be obtained. Where available, representative samples of all the constituents of the concrete used shall be taken and enclosed in heavy-duty polyethylene bags, clearly labelled and sealed or tied securely. Where such samples are not available, information on the source and nature of these materials should be given.

21.3.5 Report on sampling

One sampling report shall be required for each sample of concrete. The person responsible for taking the sample shall record that sampling was conducted according to this Section 21. The report shall include the following:

- (a) Name and location of building, structure etc.
- (b) A description of the structure or component under investigation.
- (c) The location from which the concrete sample was taken and its orientation relative to direction of casting.
- (d) The method of sampling.
- (e) Details of the concrete, in particular its age, if known.
- (f) The identification number of the sample.
- (g) The date and time of sampling.
- (h) The name and signature of the person responsible for sampling.

21.4 TREATMENT OF SAMPLES

21.4.1 Apparatus

The following apparatus shall be required:

- (a) Crushing and grinding equipment (see C1. C15.2).
- (b) Oven at 105 °C (see C1. C17.1).
- (c) Test sieves for chemical analysis (see C1. C23.1).
- (d) Airtight bottles (see C1. C24).
- (e) Concrete saw (see C1. C25).
- (f) Sample reduction device, e.g. a riffle box.
- (g) Bar magnet.

Details of the above apparatus are given in Appendix C.

21.4.2 Density

If the content of constituents determined as percentages by mass of the oven-dried concrete is to be converted to kilograms per cubic metre of concrete, the oven-dried density of a single sound piece of concrete shall first determined before any work involving breakdown of the sample.

21.4.3 Original water content

If the original water content is to be determined, a slice of approximately 20 mm thick and having a single face area of not less than 7500 mm² shall be obtained by making two parallel cuts in the sample, preferably with a diamond saw. Care shall be taken to avoid the loss of material out of the cut faces.

To reduce carbonation of the concrete, a specimen for water content determination shall be stored in an airtight container until ready for testing.

21.4.4 Nature of aggregate

The next important procedure shall be to review carefully (see C1. 21.9.2) the concrete sample, and roughly separate both the coarse and fine aggregates under the 3 major categories as stated below:

type I : natural aggregates basically insoluble in dilute hydrochloric acid.

type S : natural aggregates mostly soluble in dilute hydrochloric acid.

type O : other aggregates.

21.4.5 Preparation of test samples

21.4.5.1 Initial treatment

The sample shall be broken into lumps not larger than about 50 mm in size, avoiding as far as possible fracture of the aggregate. The lumps shall be dried in the oven at $(105 \pm 5)^\circ\text{C}$ for 15 hours to 24 hours and allowed to cool to room temperature.

The dried sample shall be divided into two portions, A and B, with portion A approximately three-quarters of the bulk. Each portion shall remain as nearly as possible representative of the whole.

21.4.5.2 Determination of the presence of PFA

Since there is no reliable method at present available to determine the cement content of hardened concrete containing PFA, it is necessary to establish whether or not PFA is present in the concrete prior to the analysis for cement content.

The method of determining whether PFA is present in the concrete is given in Clause 21.5. If PFA is found to be present, the determination of cement content is not possible and the further preparation of the portion A of the sample need not be carried out unless it is required in other tests such as the determination of chloride content.

21.4.5.3 Treatment of portion A

Inappropriate sample preparation is the biggest measurement uncertainty component throughout the testing of hardened concrete. Correct sample preparation shall include the prevention of loss of sample material, especially dust, during the entire crushing and grinding processes. Unreasonable particle size reduction shall not be practised during the preparation of the analytical sample. The crushing, grinding and subsequent processes shall be conducted as fast as possible, such that the sample shall have no time to expose to atmospheric carbon dioxide. The procedure to obtain a representative analytical sample of at least 30 g of ground material, after passing a 150 μm sieve, shall be as follows:

Using the crushing and grinding equipment, portion A shall be crushed until it all passes a 5.0 mm sieve. It shall then be subdivided, using a riffle box or by equivalent means, to produce a sub-sample of 500 g to 1000 g. This sub-sample shall be crushed to pass a 2.36 mm sieve and reduced by two separate subdividing operations, discarding one half on each occasion. The whole of the remaining part shall be ground to pass a 600 μm sieve and again reduced by two separate subdividing operations, discarding one half on each occasion. The whole of the remaining part shall be ground to pass a 150 μm sieve.

When other mechanical methods involving crushing and grinding have to be used, some of the analytical processes may be skipped. For instance, the initial crushing of portion A may yield a sample passing a 1.18 mm sieve. Then subdividing the sample at once should provide a sub-sample of 100 g to 200 g. Such sub-samples can then be grinded in a suitable grinder for passing a 150 μm sieve.

Prior to the final grinding process, it is a good practice that the resultant sieved sample (i.e. after passing a 150 μm sieve) is removed by sieving, reserved and recombined with the ground fraction.

The analytical sample so obtained shall be placed in a porcelain evaporating basin or similar container and the metallic iron removed by stirring the sample with a bar magnet. The analytical sample shall then be transferred to a clean dry bottle with an airtight closure and thoroughly mixed by tumbling, rolling or shaking for at least two minutes.

When aggregate control samples are available, they shall be dried and ground by the same method as that used for the concrete.

21.4.5.4 Treatment of portion B

Portion B shall be retained in an airtight container for use in the test described in clauses 21.8, 21.9 and 21.10.

21.5 DETECTION OF PFA

21.5.1 General

As PFA contains characteristic spherical particles, its presence in hardened concrete may be detected by microscopic examination of the residue after treatment of the concrete with dilute acid.

21.5.2 Reagents

The following reagent is required:

Dilute hydrochloric acid (1 + 9).

21.5.3 Apparatus

The following apparatus is required:

- (a) Amd No. 1/2013 75 µm sieve (see C1. C23.2).
- (b) Centrifuge (see C1. C26).
- (c) Transmitted-light microscope (see C1. C27).

Further details of the apparatus are given in Appendix C.

21.5.4 Preparation of sample

Approximately 400 g of portion B prepared in accordance with C1. 21.4.5.1 shall be obtained using a riffle box. The sample shall then be broken down in the crushing equipment, and aggregate removed as far as possible to ensure an adequate concentration of the cement matrix.

About 5 g of this cement-rich portion of size 3 to 5 mm shall be randomly selected from the bulk and transferred to a 200 mL beaker, 100 mL of the hydrochloric acid shall be added and stirred for 20 minutes with occasional breakdown of any remaining lumps using a plastic rod. The contents of the beaker shall be transferred to a centrifuge bottle which shall then be stoppered and allowed to spin in the centrifuge for five minutes. The supernatant shall be removed by decantation and the bottle containing the residue shall be refilled with 100 mL of water and then centrifuged for another five minutes.

The above process of decantation of the supernatant, washing of the residue by water and spinning shall be repeated twice before the contents of the bottle are emptied through a 75 µm sieve Amd No. 1/2013 into a dry slow filter

paper.

21.5.5 Microscopic examination

A speck of the sample residue retained on the filter paper shall be spread evenly over the centre of a microscope slide with the aid of a drop of dispersal agent and then covered by a glass cover. A second slide shall be prepared in the same manner with a speck of the sample residue taken from a different part of the filter paper.

The slides shall be examined using the transmitted-light microscope with a magnification of 200 to 400. The whole area of the sample being scanned in each case. The presence of spherical particles in either slide indicates that the concrete with and without PFA are reproduced in Figure 21.

NOTE. To avoid possible contamination, the 75 µm sieve should be washed and cleaned before use. Amd No. 1/2013

21.6 CEMENT AND AGGREGATE CONTENTS

21.6.1 General

The reported results between the cement and aggregate contents, are governed by the tests stated under this Section. Under any circumstances, representative samples of both cement and aggregate used, in making the concrete, shall be obtained. In brief, chemical analyses on cement and aggregate samples shall be conducted similarly to that described for concrete sample.

If the original constituents of concrete samples are not given (or not available) upon commencement of analysis, then the observed contents and actual contents of cement and aggregate respectively, shall rely on the correctness of the assumptions made on the composition of the materials present in the concrete sample (see C1. 21.6.6 and Table 6). For instance, an error of 1 % in the assumed calcium oxide content of an aggregate is equal to an error of about 35 kg/m³ in the calculated cement content. Likewise, an error of 1 % in the assumed soluble silica content of an aggregate is equal to an error of about 110 kg/m³ in the calculated cement content.

One or more of the methods described under this Section may be used as appropriate. In C1.21.6.5.1, there states the extraction method of soluble silica, and it is very important that this method shall be strictly adhered to without any departures/ deviations. Under this Section 21, it is allowed to use other methods that shall have been supported with proven accuracy, for analysing the following parameters:-

- (a) Calcium oxide e.g. by atomic absorption.
- (b) Soluble silica e.g. by atomic absorption.
- (c) Loss-on-ignition e.g. by thermogravimetry.
- (d) Carbon dioxide e.g. by instrumental methods.

21.6.2 Reagents

The following reagents are required:

(a) Ammonium chloride solution, 1g/L

1 g of ammonium chloride shall be dissolved in 1 L of water.

(b) Calcein (screened) indicator

The indicator shall be obtained by mixing and grinding together 0.2 g of calcein, 0.12 g of thymolphthalein and 20 g of potassium chloride.

(c) Calcium standard solution

A quantity of 1.78 g to 1.79 g of pure calcium carbonate shall be dried at a temperature in the range 150°C to 200°C and its mass recorded to the nearest 0.0001 g. It shall then be dissolved in a slight excess of dilute hydrochloric acid (1 + 4), boiled to expel carbon dioxide, cooled and diluted to 1 L in a volumetric flask.

The concentration of CaO shall be expressed in g/L to four significant figures as mass of CaCO_3 / 1.7848.

(d) Triethanolamine solution (1 + 4)

(e) Sodium hydroxide solution, 200 g/L

(f) EDTA standard solution

The solution shall be obtained by dissolving 6.67 g of EDTA (ethylenediamine-tetra-acetic acid, disodium dihydrate salt) in warm water, filtering if necessary, cooling and diluting to 1 L. The solution shall be stored in a polyethylene bottle. The solution shall be standardized against the calcium standard solution as follows:

20 mL of the calcium standard solution shall be pipetted into a 250 mL beaker or flask. 10 mL of triethanolamine solution and 10 mL of sodium hydroxide solution shall be added and the solution shall be made up to an about 150 mL with water. About 0.15 g calcein indicator shall be added and the solution titrated with the EDTA standard solution until the fluorescent green of the indicator completely changes to pink-purple with no residual fluorescence. The end-point is more easily detected if examined against a black background. The CaO equivalent of the EDTA, E , (in mg CaO/mL) shall be calculated from the expression:

$$E = \frac{20 h}{V} \quad \text{----- (21 - 1)}$$

where

h is the concentration of CaO in the calcium standard solution (in g/L)
 V in the volume of EDTA required in the titration, to the nearest 0.02 mL

(g) Concentrated hydrochloric acid, relative density 1.18.

(h) Dilute hydrochloric acid

(1 + 1), (1 + 4), (1 + 9) and (1 + 49).

(i) Polyacrylamide solution, 1 g/L

0.1 g of polyacrylamide, molecular weight about 5×10^6 shall be added to 100 mL of water and stirred using a mechanical stirrer until dissolved.

(j) Polyethylene oxide solution, 2.5 g/L

The solution shall be obtained by slowly adding 0.5 g of polyethylene oxide, molecular weight less than 1×10^6 , to 200 mL of water and stirring using a mechanical stirrer until dissolved. The solution shall be discarded after two weeks.

(k) Sodium carbonate solution, 50 g/L

The solution shall be obtained by dissolving 50 g of anhydrous sodium carbonate in 1 L of water.

21.6.3 Apparatus

The following apparatus is required:

- (a) Furnace (see C1. C28).
- (b) Desiccator (see C1. C29).
- (c) 250 mL polypropylene beakers.
- (d) Plastic stirring rods.
- (e) Water or steam bath.

Further details of the apparatus are given in Appendix C.

21.6.4 Determination of calcium oxide when insoluble residue and soluble silica are not to be determined

A quantity of (5 ± 0.1) g of the analytical sample prepared as in C1. 21.4.5.3 shall be weighed accurately to the nearest 1 mg and transferred into a 500 mL beaker and dispersed with 50 mL to 100 mL of cold water. 10 mL of concentrated hydrochloric acid shall be added, swirled and immediately made up with hot water to about 150 mL. The solution shall be brought to the boil and kept hot for five minutes. After cooling, the entire content of the beaker shall be transferred to a 500 mL volumetric flask, diluted to the mark with water and mixed thoroughly. A portion of the solution shall be filtered through a dry medium filter paper or, alternatively, a centrifuge shall be used to obtain a clear solution.

25 mL aliquot of the clear solution shall be pipetted into a 250 mL beaker or flask and

diluted to about 150 mL. 10 mL of the triethanolamine solution, 10 mL of the sodium hydroxide solution and about 0.15 g of screened calcine indicator shall be added. The solution shall then be titrated with EDTA standard solution using the magnetic stirrer until the fluorescent green of the indicator completely changes to pink-purple with no residual fluorescence (see C1. 21.6.2 (f)). The volume, V, of EDTA used in the titration shall be recorded to the nearest 0.02 mL.

The calcium oxide content D as a percentage by mass of the analytical sample shall be calculated to the nearest 0.1 %, from the expression:

$$D = \frac{2VE}{m} \quad (\text{in \%}) \quad \text{----- (21 - 2)}$$

where

- V is the volume of EDTA solution used in the titration (in mL)
- E is the calcium oxide equivalent of the EDTA solution (in mg CaO/mL)
- m is the mass of the analytical sample (in g)

21.6.5 Determination of insoluble residue, soluble silica and calcium oxide

21.6.5.1 Extraction of soluble silica and preparation of stock test solution

A quantity of (5 ± 0.1) g of the analytical sample prepared as in C1. 21.4.5.3 shall be weighed accurately and transferred into a 250 mL polypropylene beaker and 100 mL of dilute hydrochloric acid (1 + 9) carefully added. If a limestone aggregate is certain to suggest its presence, or when vigorous effervescence on adding the acid indicates its likely existence, a further 10 mL of concentrated hydrochloric acid shall be added to the stirred suspension. An appropriate magnetic stirrer shall be used, and the suspension shall be stirred at room temperature for 20 minutes. It shall then be allowed to settle and the liquid decanted through either a medium filter paper or a paper pulp pad supported on a perforated cone or similar, with or without suction. The residue in the beaker shall be washed with three 25 mL portions of dilute hydrochloric acid (1 + 49) and the washings poured through the same filter paper. This first filtrate shall be retained.

NOTE. The rate of filtration will be faster by adding 1 mL of polyacrylamide solution to the acidic extract, just before the completion of the 20 minutes stirring. Coarse filter papers can be used.

The filter paper plus any residue it contains shall be placed into the beaker containing the residue and 100 mL of the sodium carbonate solution shall be added. The beaker shall be placed on a boiling water or steam bath for 15 minutes stirring occasionally with a plastic rod to break up the filter paper.

The contents of the beaker shall be transferred on to a medium hardened ashless filter paper or a paper pulp pad supported on a perforated cone or equivalent, paying special attention to recover all the residue sticking firmly to the sides of the beaker and the stirrer blade or magnet. It shall be washed six times with the hot ammonium chloride solution, twice with hot dilute hydrochloric acid (1 + 49) and twice with hot water, the volume of each of the washings being about 25 mL. Each washing shall be allowed to drain before continuing with the next. The second filtrate and the washings shall be retained.

To minimize the likelihood of silica being added back into the solution, the filtrate should be collected in a plastic beaker.

10 mL of concentrated hydrochloric acid shall be added to the first filtrate, which shall then be carefully combined with the second filtrate and washings. The resultant mixture shall be stirred rapidly in order to prevent a precipitate to be formed, whereby such precipitate is difficult to dissolve. The combined filtrates shall be reserved for conducting the rests of other processes as stated in C1. 21.6.5.3.

21.6.5.2 Determination of insoluble residue

To determine the insoluble residue, the filter paper containing the residue shall be placed in a weighed crucible and ignited, at first slowly until the carbon of the paper is completely consumed without flaming and finally at (925 ± 25) °C until constant mass (i.e. successive weighings giving a difference in mass of less than 0.5 mg) is achieved (see C1. 21.8.3). The crucible and residue shall be allowed to cool to room temperature in the desiccator and weighed. The insoluble residue content of the analytical sample shall be calculated to the nearest 0.1 % from the expression:

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

----- (21 - 3)

21.6.5.3 Determination of soluble silica

The combined filtrates and washings shall be evaporated on a boiling water or steam bath until not more than approximately 5 mL remain. 10 mL of dilute hydrochloric acid (1 + 1), half of an ashless filter tablet and 5 mL of the polyethylene oxide solution shall be added stirring thoroughly during and after each addition. It shall be allowed to stand for five minutes and then filtered through a medium filter paper. All the solids shall be transferred to the filter, rinsing it if necessary, and washed thoroughly with hot water. The volume of the filtrate and washings shall be adjusted to 500 mL in a volumetric flask, and mixed thoroughly to give the stock test solution for treatment as described in C1. 21.6.5.4.

The filter paper containing the precipitate shall be placed in a weighed platinum crucible and ignited, at first slowly until the carbon of the paper is completely consumed without flaming and finally at (1200 ± 50) °C until constant mass (i.e. difference in mass < 0.5 mg) is achieved (see C1. 21.8.3). The crucible and residue shall be allowed to cool to room temperature in the desiccator and then weighed. The soluble silica content of the analytical sample shall be calculated to the nearest 0.1 % from the expression:

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

----- (21 - 4)

The purity of the ignited silica precipitate shall be checked by evaporating it in the crucible with 10 mL of hydrofluoric acid containing five drops of sulphuric acid (1 + 1) in a properly maintained fume cupboard. The residue shall be ignited and weighed as before. The loss in mass after evaporation and ignition shall equal to the original ignited residue mass of silica. When the two masses differ by more than 0.5 mg, it is strongly recommended that the analysis (C1. 21.6.5) be repeated. If the discrepancy still occurs, the mass of silica found by

evaporation with hydrofluoric acid shall be used to calculate the soluble silica content of the analytical sample.

21.6.5.4 Determination of calcium oxide

25 mL aliquot of the stock test solution from C1. 21.6.5.3 shall be pipetted into a 250 mL beaker or flask and diluted to about 150 mL. 10 mL of the diluted triethanolamine solution, 10 mL of the sodium hydroxide solution and about 0.15 g of screened calcein indicator shall be added. The solution shall then be titrated with the EDTA standard solution and the calcium oxide content of the analytical sample calculated as described in C1. 21.6.4.

21.6.6 Calculation of cement content

By assuming the combined water of hydration of the concrete to be $0.23 \times C_1$ and the total mass of oven dried concrete to be C_1 % cement + F % aggregate + $0.23 C_1$ % combined water of hydration, the cement content as a percentage by mass of the concrete shall be calculated to the nearest 0.1 % from the following expression using first the calcium oxide determinations and then the soluble silica determinations:

$$C_1 = \frac{c - b}{(a - 1.23b)} \times 100 \% \quad \text{----- (21 - 5)}$$

where

- a is the calcium oxide or soluble silica content of the cement (in %)
- b is the calcium oxide or soluble silica content of the aggregate (in %)
- c is the calcium oxide or soluble silica content of the analytical sample (in %)

If the calcium oxide and/or the silica contents of the cement have not been determined, the best estimate based on the values listed in Table 6 shall be used. The assumed values as well as their justification shall be reported. For the estimation of C_1 in equation (21 - 5), is recommended to use the assumed calcium oxide content, instead of the silica content as stated in Table 6. For those aggregates essentially insoluble in hydrochloric acid it may be possible to assume that the calcium oxide content is zero. For local aggregates, it may be possible to assume the calcium oxide content to be 0.2 %

and the silica content as 0.4 %.

When the two cement contents thus obtained are within 1 % (m/m) of each other, the mean value shall be reported. When the two cement contents differ by more than 1 % (m/m), the reasons for the discrepancy shall be investigated. If no reason can be found, both results shall be reported and the preferred result shall be highlighted.

Regarding the preferred reported results, factors such as analyst's experience, the age and exposure of the concrete, etc. should be considered. Testing of the sample for other parameters, such as insoluble residue, carbon dioxide, magnesium oxide, etc. may be useful when conducting an assessment. If such an assessment is not possible, the lower cement content is always preferred.

If it is known that an aggregate has a calcium oxide content of less than 0.5 %, the determination of cement content via the method of soluble silica content is not a requirement, but shall be regarded as a recommended practice, and the cement content can be based on the result of the analysis of the calcium oxide content alone.

However, if the calcium oxide content of the aggregate is 35 % or more, calculations based on calcium oxide content shall not be recommended.

If the calcium oxide content of the aggregate is 35 % or more and the soluble silica content of the aggregate is 10 % or more, the analytical sample shall be analysed for another parameter, known to be present in significantly different amounts in the cement and in the aggregate, and the cement content shall be calculated by an appropriate modification of the equation (see 21 - 5). If feasible, the parameter chosen shall basically form a greater proportion of the cement than of the aggregate.

21.6.7 Calculation of aggregate content

The aggregate content F as a percentage by mass of the concrete may be calculated to the nearest 0.1 % by using either Method 1 or 2 below:

21.6.7.1 Method 1

$$F = \frac{a - 1.23c}{a - 1.23b} \times 100 \% \quad \text{----- (21 - 6)}$$

where

- a is the calcium oxide or soluble silica content of the cement (in %)
- b is the calcium oxide or soluble silica content of the aggregate (in %)
- c is the calcium oxide or soluble silica content of the analytical sample (in %)

21.6.7.2 Method 2

NOTE. *This test cannot be used if a composite cement, e.g. Portland pozzolana cement, having a significant insoluble residue has been used.*

The aggregate content may be obtained from the following expression, which is based on the insoluble residue values of the analytical sample:

$$\text{aggregate content} = \frac{\text{insoluble residue of analytical sample}}{\text{insoluble residue of aggregate}} \times 100 \% \quad \text{----- (21 - 7)}$$

NOTE. *If samples of the aggregate are not available for analysis, it may be a little inaccurate to assume that with mostly insoluble aggregate, the percentage insoluble residue of the analytical sample (see Cl. 21.6.5.2) shall correspond to the percentage aggregate content.*

21.7 ORIGINAL WATER CONTENT

21.7.1 Applicability

21.7.1.1 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.

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

The original water contents refer to the water

present in the concrete mix at the time of setting.

The procedure involves the determination of following:

- (a) The capillary pores 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. *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 cement content.*

The water/cement ratio is expressed in two ways:

- (a) as total water/cement ratio, which includes the water absorbed by the aggregates at the time of setting.
- (b) 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.

21.7.1.2 When aggregate control samples are available

The correction for the combined water content of the aggregate shall be made.

Correcting for aggregate porosity (see C1.21.7.5.4) 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 at the time of setting. When there is some doubt about the condition of saturation of the aggregate in the set concrete, correction for aggregate porosity shall be made and only the original free water/cement ratio shall be reported. When there is no doubt, both free and total water/cement ratios shall be

reported.

21.7.1.3 When aggregate control samples are not available

The combined water of hydration of the concrete shall be assumed to be:

$$0.23 \times C_1$$

where

C_1 is the cement content of the concrete (in %) (see equation 21 – 5 under C1. 21.6.6)

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 C1. 21.7.6.2) to give the original free water/cement ratio. Otherwise no correction for aggregate porosity shall be made and only the total water/cement ratio shall be reported on the assumption that the aggregate pores were filled with water when the concrete had set.

21.7.2 Reagent

The following reagent is required:

1, 1, 1-trichloroethane.

21.7.3 Apparatus

The following apparatus is required:

- (a) Vacuum desiccator (see C1. C30).
- (b) Water pump or vacuum pump (see C1. C31).
- (c) Apparatus for the determination of combined water content (see C1. C32).

Further details of the apparatus are given in Appendix C.

21.7.4 Determination of capillary porosity

The sample shall be prepared by cutting a slice as described in C1. 21.4.3, drying it at (105 ± 5) °C for not less than 16 hours and allowed it to cool to room temperature in the desiccator.

The dried slice shall be weighed and immersed in 1,1,1-trichloroethane contained in the vacuum

desiccator.

CAUTION. *Inhalation of the vapour and contact of the liquid with the skin and eyes should be avoided. Polyethylene gloves shall be worn when handling the sample.*

The absolute pressure in the vessel shall be reduced to less than 13.5 kPa (100 mmHg) by means of the water pump or vacuum pump.

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

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

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

When the air evolution has ceased, the pressure shall be released and the sample kept immersed at atmospheric pressure for a further five minutes. The sample shall then be removed from the 1,1,1-trichloroethane. Excess superficial liquid shall be allowed to drain away and excess solvent on the surface of the sample shall be quickly wiped off. The saturated sample shall be placed in a weighed polyethylene bag immediately, sealed to prevent loss of liquid by evaporation, and weighed. The mass of 1,1,1-trichloroethane required to fill the pores of the concrete shall be calculated and the equivalent mass of water derived assuming the relative density of the 1,1,1-trichloroethane to be 1.33.

The capillary porosity Q shall be calculated to the nearest 0.1 % (m/m) from the expression:

$$Q = \frac{\text{mass of solvent absorbed}}{1.33 \times \text{mass of dried concrete sample}} \times 100 \% \quad ----- (21 - 8)$$

21.7.5 Determination of combined water and cement content

21.7.5.1 Sample preparation

The slice shall be removed from the polyethylene bag and heated to constant mass, i.e. until successive weighings after heating and cooling do not differ by more than 0.1 g, at $(105 \pm 5)^\circ\text{C}$. The slice shall be treated in accordance with the procedures described in C1. 21.4.5.3 to produce a representative analytical sample of not less than 30 g all passing a 150 μm sieve and the combined water and cement contents of the analytical sample shall be determined by the procedures detailed in C1. 21.7.5.2 to 21.7.5.5.

21.7.5.2 Determination of combined water content

Using the apparatus detailed in C1. 21.7.3, the combined water shall be determined by igniting the powdered sample at $(1000 \pm 25)^\circ\text{C}$ 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.

Residual moisture in the apparatus shall be purged by passing dried gas through the combustion tube, heated to 1000°C , for 30 minutes with an empty absorption tube in position for the first 15 minutes, replacing it by the filled tubes for the rest of the time. After 30 minutes, the source of heat shall be removed and the air flow continued for a further 15 minutes. The absorption tubes shall be removed from the apparatus and transferred to the balance case.

When the combustion tube has cooled to below 100°C , the absorption tubes shall be weighed and fitted into the train. Approximately 1 g of the sample shall be weighed accurately and placed into a combustion boat previously ignited to constant mass at 1000°C and then promptly inserted into the combustion tube from the air inlet end. The connection shall be replaced and the gas flow adjusted to about three bubbles per second at the bubbler. The sample shall be heated cautiously at first and the temperature maintained at 1000°C for 30 minutes. The source of heat shall be removed and the gas continued to pass for a further 15 minutes. The absorption tube shall be disconnected and transferred to the balance case and weighed after 15 minutes.

In order to determine the 'blank' of the apparatus, which should be less than 1 mg, the procedure shall be carried out from Amend No. 1/2013 the beginning with no sample in the combustion boat. If the blank is greater than 1 mg, the joints shall be checked to ensure that they are free of leaks. Then the blank determination shall be repeated. If a value greater than 1 mg is still obtained, the absorbents shall be replaced and the whole procedure repeated.

The combined water content X shall be calculated to the nearest 0.1 % (m/m) from the expression:

$$X = \frac{\text{gain in mass of absorption tubes - blank}}{\text{mass of sample}} \times 100\% - (21 - 9)$$

21.7.5.3 Determination of cement content

The cement content of the prepared sample shall be determined by the appropriate method as used for the analytical sample (see C1. 21.6.6).

21.7.5.4 Determination of aggregate porosity

About 1 kg of the dried coarse aggregate shall be weighed and the capillary porosity determined by the procedure given in C1. 21.7.4 but calculating it as a percentage q . For convenience, the aggregate shall be contained in a plastic mesh bag or supported on a sieve. The surface of the aggregate shall be dried before reweighing by shaking off the excess liquid and rapidly rubbing in a cloth.

21.7.5.5 Determination of aggregate combined water content

The solvent-saturated aggregate shall be heated to constant mass (as in C1. 21.7.5.1) at $(105 \pm 5)^\circ\text{C}$ and crushed to pass a 150 μm sieve. The combined water content shall be determined by the procedure given in C1. 21.7.5.2 but calculated as a percentage Y .

21.7.6 Calculation of the original water/cement ratio

21.7.6.1 Aggregate control samples available

In the absence of contrary information, the values for porosity and combined water content found for the coarse aggregate may be assumed to be 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

$$W_f = (Q - \frac{qF}{100}) + (X - \frac{YF}{100}) \quad (\text{in \%}) \quad \text{----- (21 - 10)}$$

$$W_f = Q + X - \frac{F}{100}(q + Y) \quad (\text{in \%}) \quad \text{----- (21 - 11)}$$

$$\text{original free water/cement ratio} = \frac{W_f}{C_1} \quad \text{----- (21 - 12)}$$

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

$$W_t = Q + X - \frac{YF}{100} \quad (\text{in \%}) \quad \text{----- (21 - 13)}$$

$$\text{original total water/cement ratio} = \frac{W_t}{C_1} \quad \text{----- (21 - 14)}$$

where

Q is the capillary porosity of the sample (in %) (see C1. 21.7.4)

q is the capillary porosity of the aggregate (in %) (see C1. 21.7.5.4)

X is the combined water content of the sample (in %) (see C1. 21.7.5.2)

Y is the combined water content of the aggregate (in %) (see C1. 21.7.5.5)

C_1 is the cement content of the concrete (in %) (see C1. 21.7.5.3)

F is the aggregate content of the concrete (in %) (see C1. 21.6.7)

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

$$Q - \frac{q' F}{100} \quad (\text{in \%}) \quad \text{----- (21 - 15)}$$

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

$$\text{original free water/cement ratio} = \frac{Q}{C_1} - \frac{q' F}{100 C_1} + 0.23 \quad \text{----- (21 - 16)}$$

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

$$\text{The original total water/cement ratio} = \frac{Q}{C_1} + 0.23 \quad \text{----- (21 - 17)}$$

21.8 IDENTIFICATION OF TYPE OF CEMENT

21.8.1 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. The results of typical analyses of various type of cement is given in Table 6.

It is strongly recommended that the results thus obtained should be supplemented 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.

Table 6 - Experimental Data of Cement Currently Available in HK

| | Cement testing based on BS EN 197-1:2000 % |
|------------------|--|
| CaO | 62.0 |
| SiO ₂ | 21.8 |
| MgO | 1.7 |

NOTE. The above CaO content in cement is subject to a variation of approximately $\pm 2.8\%$, whereas the above SiO₂ content in cement is subject to a variation of approximately $\pm 7.8\%$.

NOTE. Variations in the clinkers used in their relative proportions may cause large deviations from the above stated values.

21.8.2 Separation and analysis of matrix

A solid piece of the concrete sample shall be carefully broken down, for example by a compressive strength testing machine, and fine material shall be obtained by sieving using a 75 µm sieve. If insufficient sample is obtained, further breakdown is necessary.

The fine material so obtained shall be analysed for insoluble residue and loss-on-ignition (see C1. 21.8.3) and, or example, soluble silica (see C1. 21.6.5.3), calcium oxide (see C1. 21.6.5.4), alumina, ferric oxide, magnesium oxide, sulphuric anhydride, etc. It may be assumed that the insoluble residue represents aggregate in the fine material and the loss-on-ignition represents hydration and carbonation of the cement and the other analytical results shall be corrected by multiplying them by

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

The results shall be compared with typical analyses of various types of cement (see Table 6).

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

21.8.3 Determination of loss-on-ignition

One to two grams of the analytical sample as prepared in C1. 21.4.5.3 shall be weighed accurately into a crucible which has been previously ignited and weighed accurately. The crucible shall be covered with a lid and placed in a furnace, the temperature of which shall be raised slowly to (925 \pm 25) °C. After five minutes at this temperature, the lid shall be removed and the crucible left in the furnace at (925 \pm 25) °C for a further 30 minutes. The crucible shall be allowed to cool in the desiccator to room temperature and weighed.

This process of ignition, cooling and weighing shall be repeated until constant mass is achieved as shown by successive weighings giving a difference in mass of less than 0.5 mg. The loss-on-ignition of the analytical sample shall be calculated to the nearest 0.1 % from the following expression:

$$L_i = \frac{\text{mass of analytical sample} - \text{mass of ignited sample}}{\text{mass of analytical sample}} \times 100\% \quad (21 - 18)$$

where

L_i is the loss-on-ignition

21.8.4 Examination under a microscope

21.8.4.1 General

To establish whether ordinary Portland cement or sulphate resisting Portland cement has been used in the concrete, the usual method is examination under a microscope. To do this, at least ten anhydrous relics of cement grains greater than 40 µm in size, or at least 20 grains greater than 20 µm in size shall be examined.

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

21.8.4.2 Materials

The following materials are required:

(a) Mounting resin

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

(b) Silicon carbide powder, medium grade, e.g. 26 µm

(c) Non-aqueous liquid, e.g. kerosene

(d) Diamond paste, of grades 14 µm, 6 µm, 3 µm, 1 µm and 0.25 µm

(e) Non-aqueous solvent, e.g. acetone

(f) Potassium hydroxide solution, 10 %

10 g of potassium hydroxide shall be dissolved in 100 mL of water.

(g) Methylated spirits or ethanol

(h) Hydrofluoric acid, 40 % relative density 1.13

21.8.4.3 Apparatus

The following apparatus is required:

- (a) Vacuum desiccator (see C1. C30).
- (b) Rotary lap-plate (see C1. C33).
- (c) Reflected-light microscope (see C1. C34).
- (d) Cylindrical mould (see C1. C35.1).

Further details of the apparatus are given in Appendix C.

21.8.4.4 Preparation of the sample

The sample shall be the portion B, obtained in accordance with C1. 21.4.5.1.

The concrete sample shall be broken up and at least ten nominal 5 mm sized pieces of cement-rich matrix shall be selected as the test portion. The test portion shall be dried at a temperature not exceeding 105 °C, allowed to cool and then embedded in a low viscosity mounting resin by casting into a cylindrical mould and curing in the vacuum desiccator at the temperature recommended by the resin manufacturer.

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

The cylindrical casting shall be sectioned with a diamond saw lubricated with a non-aqueous liquid so that cut surfaces of the pieces are exposed on the sawn surface, and washed immediately using a non-aqueous solvent, e.g. acetone. The sawn surface shall be ground with silicon carbide powder lubricated with a non-aqueous liquid such as kerosene, or other suitable oil, to remove saw marks. All traces of grinding powder shall be removed.

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

The sample shall be polished carefully with progressively finer diamond paste in an oil

soluble base on a rotary lap-plate with a suitable surface. After a final polish with 0.25 µm diamond compound, the surface shall be cleaned using a non-aqueous cleaning agent and dried 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. 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.

21.8.4.5 Cement grain identification

The polished surface shall be examined through a reflected-light microscope using magnifications up to 800. If insufficient grains of anhydrous cement are found either further polished samples as described in C1. 21.8.4.4 shall be prepared so that the required number of grains can be assessed, or the method shall be reported as not applicable.

NOTE. 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. 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) The polished surface shall be immersed in the potassium hydroxide solution at 30°C for 10 to 20 seconds, then washed immediately in industrial methylated spirits or ethanol. 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) The polished surface shall be exposed to hydrofluoric acid vapour for two to five seconds 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. It is sometimes difficult to distinguish these two compounds particularly at low magnification, i.e. less than 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.

With the use of the compositional information given in C1. 21.8.4.6, and making reference to the control specimens if prepared, the apparent type of cement represented shall be identified by examining each anhydrous grain. 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, the whole procedure shall be repeated and the combined findings considered. If the dominant cement type represents fewer than Amd No. 1/2013 80 % of the grains examined overall, the cement type shall be reported as 'uncertain'.

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

Photomicrographs of ordinary Portland cement and sulphate resisting Portland cement are reproduced in Figure 22.

21.8.4.6 Mineral phase compositions of different types of cement

Portland cements not having a specification limit for tricalcium aluminate content, generally have a volume ratio of ferrite to tricalcium aluminate of less than 2:1.

21.9 IDENTIFICATION OF TYPE OF AGGREGATE

21.9.1 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, it shall be referred to a petrographer. The petrographical procedures required are outside the scope of this Standard.

21.9.2 Procedure

A broken, or preferably a sawn surface of the sample shall be prepared (see C1. 21.4.5.3). The general character of the aggregate shall be identified and the aggregate exposed shall be compared with known samples whenever possible.

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

21.10 OTHER CONSTITUENTS

21.10.1 General

The analytical sample prepared as in C1. 21.4.5.3 may be used for the determinations of chloride, sulphate and carbonate contents. 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 advantage in all these tests.

A suitable procedure is to break the concrete sample and to obtain 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 fine materials. The cement content of this separated sample has to be determined using the procedure described in C1. 21.6.6.

21.10.2 Determination of chloride content

Alternative methods based on potentiometric titration are acceptable.

21.10.2.1 Reagents

The following reagents are required:

- (a) **Nitric acid**, relative density 1.4
- (b) **Silver nitrate standard solution**, 0.1 mol/L
Powdered silver nitrate shall be dried at 150 °C for two hours, cooled in a desiccator and a quantity of 16.989 g shall be dissolved in water and diluted to 1 L. The solution shall be stored in an opaque glass bottle and protected from prolonged exposure to light.
- (c) **Thiocyanate standard solution**, approximately 0.1 mol/L
7.6 g of ammonium thiocyanate or 9.7 g of potassium thiocyanate shall be dissolved in water and diluted to 1 L. The solution shall be standardized against the silver nitrate standard solution using iron III indicator, once a week or each time a determination of chloride content is made, if less frequent.
- (d) **Iron III indicator solution**
50 g of ammonium ferric sulphate shall be dissolved in 60 ml of warm water. 10 mL of nitric acid shall be added to the solution, which is then cooled and stored in a glass bottle.
- (e) **3,5,5-trimethylhexanol (nonyl alcohol)**

21.10.2.2 Procedure

(5 ± 0.1) g of the analytical (see C1. 21.4.5.3) or separated (see C1. 21.10.1) sample shall be weighed accurately into a stoppered 500 mL conical flask. It shall be dispersed with 50 mL of water and 10 mL of the nitric acid shall be added. 50 mL of hot water shall be added, boiled for four to five minutes and kept warm for 10 to 15 minutes. (If the supernatant liquid is turbid, it shall be filtered through a fast hardened ashless filter paper and washed with hot water.) The solution shall be cooled to room temperature and a measured excess of the silver nitrate

standard solution added. 2 mL to 3 mL of 3,5,5-trimethylhexanol shall be added to the solution, the flask shall be stoppered and shaken vigorously to coagulate the precipitate. 1 mL of iron III indicator solution shall be added and titrated with the thiocyanate solution to the first permanent red colour.

The chloride ion content J of the cement shall be calculated to the nearest 0.01 % (m/m) from the expression:

$$J = \frac{(V_1 - V_2 M)}{0.1} \times \frac{0.3545}{m} \times \frac{100}{C_1} \% \quad \text{--- (21 - 19)}$$

Where

- m is the mass of sample used (in g)
- V_1 is the volume of 0.1 M silver nitrate solution added (in mL)
- V_2 is the volume of thiocyanate solution used (in mL)
- M is the molarity of the thiocyanate solution (in mol/L)
- C_1 is the cement content of the sample used (in %)

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

21.10.3 Determination of sulphate content

21.10.3.1 Reagents

The following reagents are required:

- (a) **Concentrated hydrochloric acid**, relative density 1.18
- (b) **Dilute hydrochloric acid (1 + 49)**
- (c) **Ammonium hydroxide solution (1 + 1)**
- (d) **Barium chloride solution, 100 g/L**

21.10.3.2 Procedure

(5 ± 0.1) g of the analytical (see C1. 21.4.5.3) or separated (see C1. 21.10.1) sample shall be weighed accurately into a 400 mL beaker, dispersed with 50 mL of water and 10 mL of concentrated hydrochloric acid added. If effervescence is considerable, the beaker shall be covered immediately. 50 mL of hot water shall

be added, the beaker covered and the solution boiled gently for 5 to 10 minutes. The content of the beaker shall then be filtered through a medium ashless filter paper and the residue thoroughly washed with hot dilute hydrochloric acid (1 + 49). Three drops of the methyl red indicator shall be added to the filtrate which shall then be heated to boiling and just neutralized to yellow with the dilute ammonium hydroxide solution. 1 mL of concentrated hydrochloric acid shall be added immediately and then 10 mL of the barium chloride solution shall be added dropwise. 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.

The solution shall be boiled gently for five minutes, kept as just below boiling for 30 minutes and allowed to stand at room temperature for 12 to 24 hours. It shall then be filtered through a slow ashless filter paper and washed free from chlorides with hot water. The paper and contents shall be transferred to a weighed silica or platinum crucible and the paper burnt off without flaming. The precipitate shall be ignited at 800 °C to 900 °C until constant mass is achieved (see C1. 21.8.3).

The sulphate content G , expressed as SO₃ shall be calculated, as a percentage of the cement to the nearest 0.1 % (m/m) from the expression:

$$G = \frac{B}{m} \times 34.3 \times \frac{100}{C_1} \% \quad \text{----- (21 - 20)}$$

Where

- B is the mass of ignited barium sulphate (in g)
- m is the mass of the sample used (in g)
- C_1 is the cement content of the sample used (in %)

NOTE. *The stock test solution (see Cl. 21.6.5.3) may be used for this determination. A 200 mL aliquot shall be used and the procedure as above followed from the heating stage to boiling and neutralizing with dilute ammonium hydroxide solution.*

21.10.4 Determination of carbonate

The carbonate content of the concrete may be calculated from its carbon dioxide content, two

methods of determining which are given below:

21.10.4.1 Method 1

21.10.4.1.1 Reagents

- (a) **Orthophosphoric acid**, relative density 1.7
- (b) **Concentrated sulphuric acid**, relative density 1.8
- (c) **Anhydrous copper sulphate on pumice**

50 g of pumice stone (particle size of about 1 mm) shall be added to 50 mL of 30 % copper sulphate solution (15 gm copper sulphate pentahydrate dissolved in 50 mL distilled water). The mixture shall be heated on top of a boiling water bath to near dryness with constant stirring. The mixture shall be dried in an oven at 105 °C for two hours and kept in an air-tight bottle.

- (d) **Dried magnesium perchlorate**, particle size 0.7 - 1.18 mm (10 - 16 mesh)
- (e) **Granular absorbent for CO₂, soda lime**, particle size 1.0 - 1.7 mm (10 - 16 mesh)

21.10.4.1.2 Apparatus and procedure

The apparatus as shown in Figure 27 (see C1. C36 of Appendix C) shall be assembled without the weighable tubes and flushed with gas free from carbon dioxide at a rate of approximately three bubbles per second for 15 minutes. The tap funnel shall be acid-free. Approximately 0.2 g of the analytical sample prepared as in C1. 21.4.5.3 shall be accurately weighed into a duplicate reaction flask, the empty reaction flask shall be replaced by the one containing the sample and the passing of gas free from carbon dioxide continued. The two absorption tubes shall be weighed and connected to the apparatus. The gas inlet shall be disconnected and 30 mL of orthophosphoric acid shall be placed into the tap funnel.

The tap shall be opened and the gas inlet reconnected; the gas pressure will force the acid into the reaction flask. After effervescence has ceased, the contents of the flask shall be slowly heated to boiling and boiled for five minutes. It shall then be allowed to cool for

15 minutes while maintaining the gas flow. The absorption tubes shall be detached, transferred to the balance case and weighed after 30 minutes.

To determine the 'blank' of the apparatus, which should be less than 1 mg, the procedure described above shall be carried out with no sample in place. If the blank is greater than 1 mg, the joints shall be checked to ensure that they are free of leaks. The blank determination shall then be repeated. If a value greater than 1 mg is still obtained, the absorbents shall be replaced and the whole procedure repeated.

The carbon dioxide content, C of the analytical sample shall be calculated to the nearest 0.1 % from the expression:

$$C = \frac{\text{gain in mass of absorption tubes - blank}}{\text{mass of analytical sample}} \times 100 \% \quad (21 - 21)$$

21.10.4.2 Method 2 (see Figure 28 of Appendix C)

This method is based on a manometric measurement of the volume of carbon dioxide evolved upon acid treatment of a weighed powdered concrete sample. The carbonate content is calculated from the volume of the gas after being corrected for temperature and atmospheric pressure.

21.10.4.2.1 Reagents

The following reagents are required:

(a) dilute hydrochloric acid solution (1 + 1)

(b) Methyl red indicator solution

1 g of methyl red shall be dissolved in 600 mL of methylated spirits and 400 mL of water added.

21.10.4.2.2 Apparatus

The following apparatus is required:

- (a) A graduated burette.
- (b) A plastic measuring cylinder.
- (c) A levelling tube.
- (d) A thermometer.
- (e) A barometer.

Further details of the apparatus are given in C1. C37 of Appendix C.

21.10.4.2.3 Procedure

The apparatus shall be set up as shown in Figure 28 (see C1. C37 of Appendix C). The graduated burette and levelling tube shall be filled with water to which two drops of the hydrochloric acid and a few drops of methyl red solution have been previously added.

The quantity of the analytical sample required is such as to give off 30 mL - 60 mL of carbon dioxide gas upon acid treatment. As a guide, this amount will range from 0.5 g - 1.0 g for a heavily carbonated sample to about 3 g - 5 g for one having a low carbonate content. The sample shall be weighed to an accuracy of 0.5 mg, placed in the reaction flask and about 20 mL of water shall then be added.

10 mL of the hydrochloric acid shall be put in the plastic measuring cylinder which shall then be carefully placed in the reaction flask without spilling the acid. The reaction flask shall be connected to the apparatus by inserting the rubber stopper.

With the 3-way tap opened to atmosphere, the level of the coloured solution in the graduated burette shall be adjusted until it coincides with the zero mark by raising or lowering the levelling tube. The tap shall then be closed and the reaction flask tilted so that the acid in the measuring cylinder spills over the sample. The flask shall be shaken well to ensure the sample and the acid are mixed thoroughly. The tap shall be set to the position in which the burette is connected to the flask.

The height of the levelling tube shall be adjusted until the levels of the solution in the burette and levelling tubes are equal. The tap shall then be closed and the volume displaced by the gas in the burette noted.

The flask shall be reshaken and the procedure repeated until there is no further increase in the gas volume indicated by the burette. The final volume shall be recorded as the volume of carbon dioxide evolved.

The ambient temperature and the barometric

pressure at the time of the test shall be recorded.

21.10.4.3 Calculation of carbon dioxide content

The carbon dioxide content C of the analytical sample shall be calculated to the nearest 0.1 % from the expression:

$$C = \frac{0.0705 \times V(P - W)}{(273 + T)m} \quad \text{----- (21 - 22)}$$

where

- V is the final gas volume in mL.
- P is the barometric pressure in mmHg, see Table 7
- W is the water vapour pressure (mmHg) at T °C
- T is the ambient temperature in °C
- m is the mass of sample of g

The carbonate content C' in percentage by mass of the sample may be obtained from the following expression:

$$C' = \frac{15C}{11} \quad \text{----- (21 - 23)}$$

When the carbonate content is calculated as calcium carbonate content, the following expression may be used:

$$\text{CaCO}_3 = \frac{25C}{11} \quad \text{----- (21 - 24)}$$

The results shall be reported to the nearest 0.1 %.

NOTE. Correction for the volume of acid solution should be made to the final gas volume if the acid solution is added externally to the reaction flask.

NOTE. The value of water vapour pressure W at various temperatures is shown in Table 7.

Table 7 - Vapour Pressure of Water at Temperatures between 20 °C and 30 °C

| Temperature °C | Vapour Pressure mm Hg |
|-------------------|--------------------------|
| 20 | 17.530 |
| 20.5 | 18.085 |
| 21 | 18.650 |
| 21.5 | 19.231 |
| 22 | 19.827 |
| 22.5 | 20.440 |
| 23 | 21.068 |
| 23.5 | 21.714 |
| 24 | 22.377 |
| 24.5 | 23.060 |
| 25 | 23.756 |
| 25.5 | 24.471 |
| 26 | 25.209 |
| 26.5 | 25.964 |
| 27 | 26.739 |
| 27.5 | 27.535 |
| 28 | 28.349 |
| 28.5 | 29.184 |
| 29 | 30.043 |
| 29.5 | 30.923 |
| 30 | 31.824 |

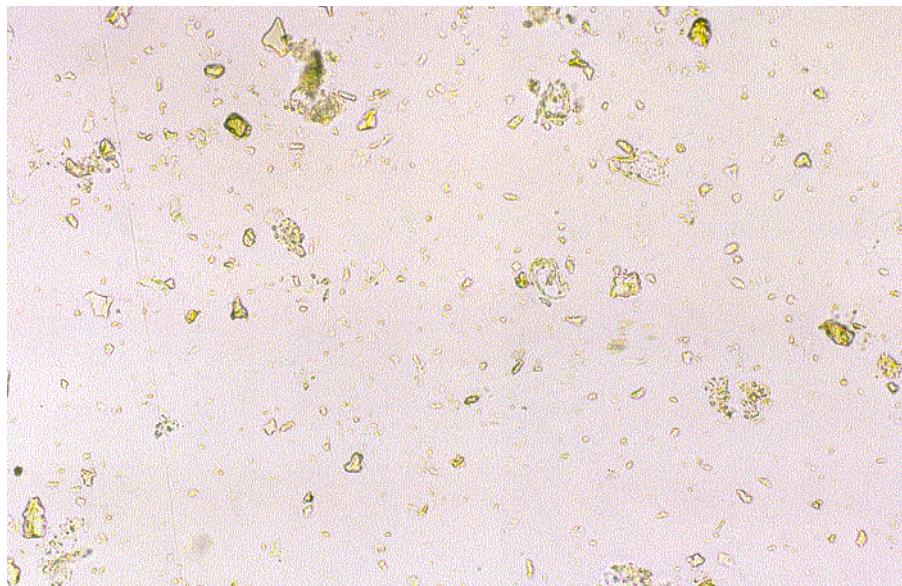
21.11 REPORT

The report shall affirm that the analysis was made in accordance with this Standard. When alternative methods are used, the report shall include reference to evidence held by the laboratory showing that the alternative methods are capable of producing results which do not differ significantly from those obtained by using this the Standard method. A copy of the sampling report shall be provided with this report.

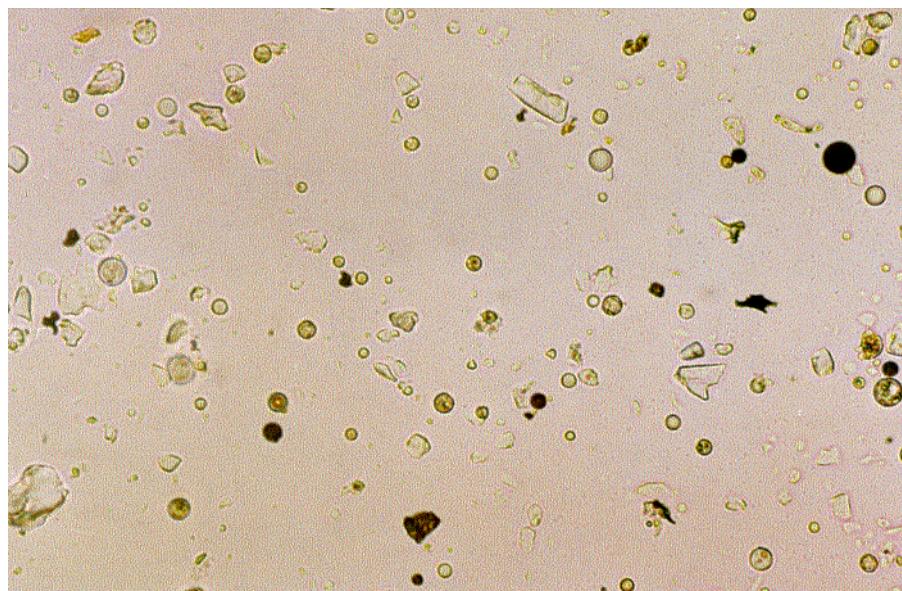
The report shall include the following information:

- (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) Any assumptions made in the analysis, e.g. type of aggregate, cement analysis, aggregate analysis, etc..

- (e) Reporting of individual test results, and the average test result if applicable, after the completion of each chemical analysis.
- (f) Any other results obtained coincidentally to the tests required.
- (g) The results of any additional tests done at the analyst's discretion.
- (h) Name and signature of person responsible for the test.



OPC Concrete (Magnification: 200X)

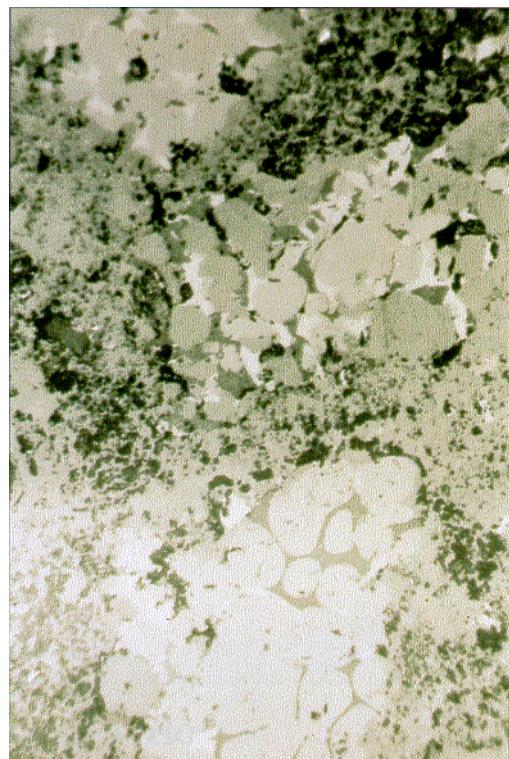


Concrete Containing PFA (Magnification: 200X)

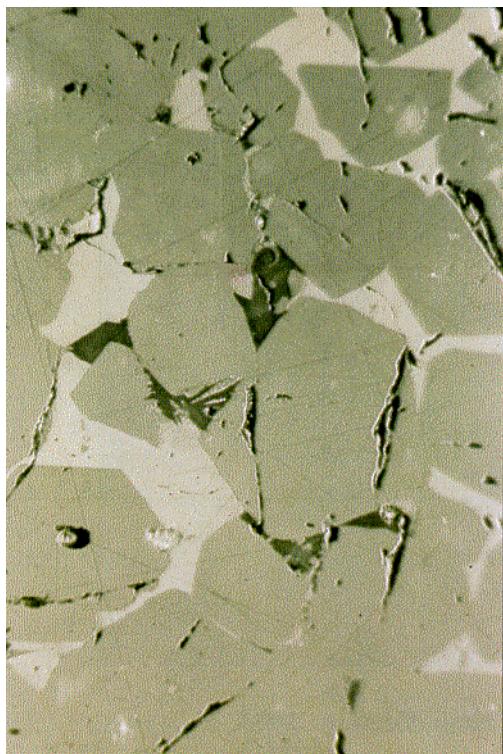
Figure 21 - Photomicrographs of OPC and PFA Concrete



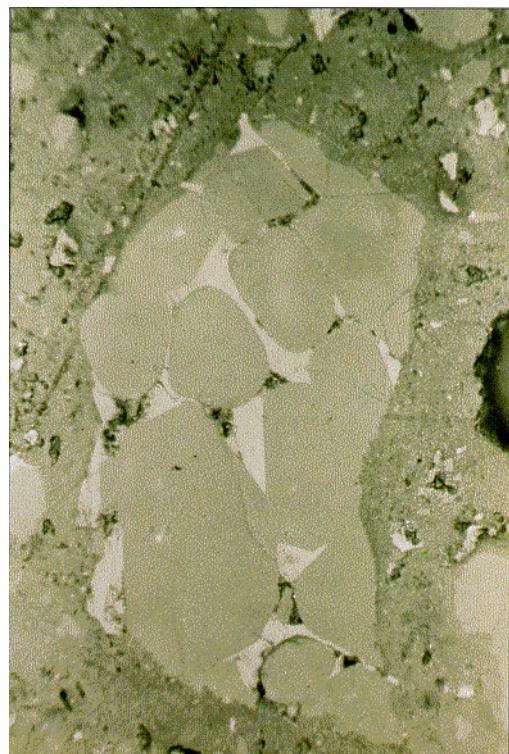
Ordinary Portland Cement (OPC)



OPC Concrete



Sulphate Resisting Portland Cement (SRPC)



SRPC Concrete

Figure 22 - Photomicrographs of OPC and SRPC Cement/Concrete

SECTION 22

DETERMINATION OF ALKALI SILICA REACTION POTENTIAL BY ULTRA-ACCELERATED MORTAR BAR TEST

22.1 SCOPE

This test method is intended to determine rapidly the potential alkali-reactivity of aggregates that forms the main constituents of concrete through the evaluation of the expansion of mortar-bars immersed in NaOH solution at elevated temperature, as specified in the method. The test may also be used in experiments to assess the pessimum behaviour of reactive aggregates.

NOTE. *With some reactive aggregates it has been found that there is a proportion of reactive constituents in the aggregate that leads to maximum expansion. This proportion is called the "pessimum content" and the relationship between expansion and reactive constituents content is called the "pessimum behaviour" of the reactive aggregate.*

It is recommended to start all screening of aggregates for their potential alkali-reactivity with a petrographic examination of the aggregate. On the basis of the results of the petrographic examination, a decision can be made with respect to further testing. If aggregates contain more than 2 % (by mass) of porous chert and flint, further testing by means of the accelerated mortar-bar test is not recommended as sometimes it gives rise to misleading results and inappropriate approval of such aggregates.

22.2 APPARATUS

The following apparatus is required :

- (a) Mixer for mortar bar test (see Cl. C1.2).
- (b) Containers for mortar bar test (see Cl. C2.3).
- (c) Balance (see Cl. C8.1 & C8.3).
- (d) Crushing and grinding equipment (see Cl. C15.2).
- (e) Oven at 80 °C (see Cl. C17.2).
- (f) Test sieves for mortar bar test (see Cl. C23.3).
- (g) Mould for flow test (see Cl. C35.2).
- (h) Prism moulds for mortar bar test (see Cl. C35.3).
- (i) Flow table (see Cl. C38).
- (j) Tamper (see Cl. C39).
- (k) Length comparator (see Cl. C40).
- (l) Storage cabinet/temperature controlled cabinet (see Cl. C41.1).
- (m) Thermometer (see Cl. C42).
- (n) Sample divider - Device for sample reduction such as a riffle box.

Further details of the apparatus are given in Appendix C.

22.3 REAGENTS

22.3.1 Water

Distilled or deionised water.

22.3.2 Sodium hydroxide solution (NaOH)

Sodium hydroxide (NaOH) shall be at least technical grade. Each litre of sodium hydroxide solution shall contain 40.0 g of NaOH dissolved in 900 ml of water and, after cooling to about 20 °C, it shall be diluted with additional distilled or deionised water to obtain 1.0 litre of solution. The concentration of the solution shall lie between 0.99 and 1.01 M. A new solution shall be prepared for each series of tests.

NOTE. *Reliable safety precautions should be taken and suitable personal protective equipment should always be used to avoid the hazards of the hot alkaline solution such as severe burns and injury to unprotected skin and eyes.*

22.4 MATERIAL

Portland cement CEM1 or ASTM Type 1 with a minimum Na₂O equivalent (Na₂O + 0.658 K₂O) of 1.0 %. The specific surface of the cement, when measured according to the air permeability method (e.g. BS EN 196-6:2010 Amd No. 1/2013), shall be greater than 450 m²/kg. The autoclave expansion, determined according to ASTM test method C151 shall be less than 0.20 %. Alternatively the MgO soundness can be evaluated by Le Chatelier test (e.g. BS EN 196-3:2005+A1:2008 Amd No. 1/2013) and the increase in separation of indicators ends shall be 0 mm.

NOTE. *Suitable reference cements are available from Norcem A.S, R & D Department, 3950 Brevik, Norway, or from National Council for Cement and Building Materials, Calibration Testing & Quality Control, 34 Km Stone, Delhi-Mathura Road (NH-2), Ballabgarh 121 004, Haryana State, India.*

22.5 PREPARATION OF THE AGGREGATE SAMPLE

22.5.1 Natural fine aggregate

The natural material proposed for use as fine aggregate in concrete shall be tested in the grading as submitted, except that particles retained on a 4 mm screen or equivalent shall be crushed and included.

NOTE. *The particles retained on the 4 mm screen or equivalent shall be removed only if petrography has confirmed that they are no different from the particles passing through.*

22.5.2 Crushed fine aggregate

The crushed material proposed for use as fine aggregate in concrete shall be tested in the grading prescribed in Table 8.

Table 8 - Grading Requirements

| Sieve size | | Mass (%) |
|-------------|-------------|----------|
| Passing | Retained | |
| 4 mm | 2 mm | 10 |
| 2 mm | 1 mm | 25 |
| 1 mm | 500 μ m | 25 |
| 500 μ m | 250 μ m | 25 |
| 250 μ m | 125 μ m | 15 |

NOTE. *Alternatively the test sieves with supplementary sizes as described in Cl. C23.3 can be used.* Amd No. 1/2013

22.5.3 Coarse aggregate

The material proposed for use as coarse aggregate in concrete shall be processed by crushing and sieving to produce a graded sample in accordance with the requirements prescribed in Table 8. This sample shall be representative of the composition of the coarse aggregate as proposed for use.

NOTE. *Coarse aggregate crushed to sand size may give increased expansion, owing to the increased surface exposed upon crushing. Therefore, if coarse aggregate tested by this method is found to be potentially reactive, tests should be performed on concrete specimens, according to section 23 of this Standard (The Concrete Prism Test) to verify the results found with the mortar samples.*

22.5.4 Fine and coarse aggregate

The material proposed for use as fine and coarse aggregate in concrete shall be separated by sieving on a 4 mm sieve or equivalent. Fine and coarse aggregate shall be tested separately as indicated in clauses 22.5.1, 22.5.2 and 22.5.3.

22.5.5 Final aggregate preparation

After the fine or coarse aggregate has been processed and washed to remove adhering dust or fine particles ($< 125 \mu\text{m}$) from the aggregate, the fractions shall be dried at 100 °C to 110 °C for 16 hours and cooled. Unless used immediately, each fraction shall be stored individually in a clean container provided with a tight-fitting cover.

22.5.6 Combining the aggregates

If the test method is to be used in an experimental programme to assess the pessimum behaviour of a reactive aggregate, a petrographic examination of the test sample shall be undertaken according to RILEM Recommended Test Method AAR-1: Detection of potential alkali-reactivity of aggregates – Petrographic method Amd No. 1/2013. This examination should be sufficiently detailed to identify all the rock types or mineral constituents that compose the test sample. The results from the petrographic examination could then be used to establish a suitable test protocol as listed below such that any potentially damaging pessimum behaviour is identified.

If the aggregate sample is composed of,

- (a) particles of a single rock, sand, gravel or mineral type that has previously been shown not to have an associated pessimum behaviour and it is intended to use this aggregate unblended, or
- (b) the reactive constituent in a single rock, sand, gravel or mineral type is found at concentrations outside the pessimum limits previously established for the reactive constituent type and it is intended to use this aggregate unblended,

then only a mortar with an aggregate component

composed wholly of the test aggregate needs to be tested.

Alternatively, if an aggregate sample is,

- (a) composed wholly or in part of a rock, sand, gravel or mineral type which has particles or mineral constituents that have an associated pessimum proportion and the reactive constituents are found in concentrations that are deemed to be potentially reactive, or it is either unknown or not prescribed where the "safe" limits lie, or
- (b) if it has not been established whether the rock or mineral constituent type has or does not have an associated pessimum proportion, or
- (c) if the sample is a blend of aggregate types where the behaviour of the aggregates when combined is unknown, or
- (d) if a petrographic analysis is not available,

then several combinations of test aggregate and reference non-reactive fine material will need to be tested, as indicated in the following paragraph, in order to identify any pessimum behaviour.

The aggregate to be tested shall be mixed with a reference non-reactive fine material (natural sand or crushed rock) with the same grading as the sample under test. The material used for this purpose shall give an expansion of less than 0.05 % by this test method (see note below) and shall not exhibit a pessimum behaviour. It should be preferably a non-siliceous material and previously evaluated. Some suggested proportions of test aggregate and reference non-reactive fine material are shown in Table 9. As preliminary tests, only three combinations should be used (e.g. I, II and IV). Special aggregates may require some other levels to make sure that any possible pessimum behaviour is identified.

NOTE. This expansion limit is deliberately more restrictive than is generally applied to identify non-reactive aggregates to ensure that the non-reactive reference aggregate has minimal effect on the results of this test.

Table 9 - Suggested Aggregate Proportions

| Combination | Aggregate to be tested (%) | Reference non-reactive fine material (%) |
|-------------|----------------------------|--|
| I | 100 | 0 |
| II | 50 | 50 |
| III | 25 | 75 |
| IV | 15 | 85 |
| V | 5 | 95 |
| VI | 0 | 100 |

22.6 CONDITIONING

The temperature of the moulding room, apparatus, dry materials, mixing water and of the cabinet or moist chamber shall be maintained at (20 ± 2) °C. The relative humidity of the moulding room and of the cabinet or moist chamber shall not be less than 60 % and 90 % respectively.

22.7 PROPORTIONING OF MORTAR

22.7.1 Proportion of mortar

The dry materials for the test mortar shall be proportioned using one part of cement to 2.25 parts of aggregate by mass. The minimum quantity of dry materials to be mixed at one time for making three 25 mm x 25 mm x 285 mm specimens shall be 400 g of cement and 900 g of aggregate.

22.7.2 Workability of mortar

A free water/cement ratio of 0.47 by mass shall be used, where the free water is the water available for hydration of cement and for the workability of the fresh mortar. The total water added to the mix is the free water plus the water absorbed by the aggregate to bring it to a saturated surface dry condition. The workability of the mortar shall be measured on a flow table using the procedure described in BS 4551-1:1998 Amd No. 1/2013 with the exception that the flow table shall be raised and dropped 10 times during a period of 6 s through a height of (12.7 ± 0.1) mm. The flow value (mean diameter of the mortar) shall be in the range of 205 to 220 mm and, in the cases of mixes with a flow initially less than 205 mm, a Amd No. 1/2013

superplasticizer may be used to achieve the required flow but the superplasticizer shall not contain air entraining agent.

22.8 PROCEDURE

22.8.1 Moulding test specimens

At least three 25 mm x 25 mm x 285 mm specimens shall be made out of each one of two batches of mortar for every aggregate to be tested.

The mould shall be prepared with a suitable releasing agent that will not affect the setting of the cement or leave any residue that will inhibit the penetration of water into the specimen. Two gauge studs shall be fixed in each mould so that they align with the principal axis of the test specimen. The mortar shall be mixed in accordance with the procedure for testing the strength of cement with plastic mortar (e.g. BS EN 196-1:2005 Amd No. 1/2013). The moulding of specimens shall commence not more than 3 minutes after completion of the mixing of the mortar. The moulds shall be filled with two approximately equal layers, each layer being compacted with the tamper. The mortar shall be worked along the surface of the mould with the tamper until a homogeneous specimen is obtained, making sure that the mortar is fully pushed under the reference inserts before a second layer is placed into mould. After the top layer has been compacted, the mortar shall be levelled with the top of the mould and the surface shall be trowelled smooth with a few strokes of the trowel.

NOTE. Particular care should be taken to obtain a consistent compaction of the mortar, as the degree of compaction greatly influences the degree of expansion, the better the compaction the lower the expansion.

22.8.2 Initial Curing and Measurement

22.8.2.1 Curing prior to demoulding

The moulded specimens shall be placed in the moist chamber for a period of (24 ± 2) hours. The specimens shall be demoulded protected from loss of moisture. Each specimen shall be properly identified in such a way that they, when subsequently measured, are placed in the measuring equipment in the same manner. One end of each beam shall be designated as the top and this end shall be marked with a number for

identification. This end shall be kept uppermost during all subsequent measurements. The reference length (L_i) of the demoulded beam shall be measured and recorded to the nearest 0.002 mm.

22.8.2.2 Curing after demoulding

The specimens made with each aggregate sample shall be placed in a storage container with sufficient distilled or deionized water, at room temperature and the specimen shall be totally immersed. The containers shall then be sealed and placed in an oven at (80 ± 2) °C for a period of 24 hours.

22.8.2.3 Taking zero measurement (L_0)

The containers shall be removed from the oven one at a time. Other containers shall be removed only after the bars in the first container have been measured and returned to the oven. The bars shall be removed one at a time from the water and their surface shall be dried with a towel or cloth paying particular attention to the two metal pins. The zero measurement of each bar (L_0) shall be taken immediately after drying and read as soon as possible after the bar is in position. The process of drying and measuring shall be completed within 15 s of removing the specimens from the water.

NOTE. The measuring device should be checked with the reference bar prior to and after measurement of each set of specimens. If the difference between the initial and final measurements on the reference bar exceeds 3 µm, all the measurement shall be repeated. The ends of the reference rod, beam studs and the contact surfaces of the comparator shall be cleaned prior to taking a comparator reading. Also, the rod or the bar shall be slowly rotated relative to the measuring device while reading is being taken in order to achieve a consistent result. If the rotation caused a small change in the gauge reading, the minimum reading of the gauge shall be recorded.

22.8.3 Final Storage and Measurement

22.8.3.1 Final storage

The specimens made with each aggregate sample shall be immersed in 1 M NaOH solution, preheated at (80 ± 2) °C. The recommended volume proportion of sodium hydroxide solution to mortar-bars in the storage container shall be (4 ± 0.5) times the volume of

the mortar-bars. The container shall be sealed and returned to the oven.

22.8.3.2 Subsequent measurement (L_n)

Subsequent measurements (L_n) of the specimen shall be taken periodically, with a reading after 24 hours of immersion in the NaOH solution and at least three intermediate readings before the final reading at 14 days of immersion in NaOH solution. If so desired, measurement may be taken at 24-hour intervals and may be continued beyond 14 days. All measurements should be taken at approximately the same time each day. The measuring procedure is identical to that described in clause 22.8.2.3 and the specimens shall be returned to their container after each measurement.

NOTE. *In some cases, the solution may become cloudy due to the presence of alkali-silica gels.*

22.9 CALCULATION AND REPORTING OF RESULTS

22.9.1 Expansion

The linear expansion of each specimen shall be obtained by calculating the difference between the length of the specimen at each period of measurement (L_n) and the zero measurement (L_0), to the nearest 0.001 % of the effective length, as follows :

$$\text{Expansion (\%)} = 100 \times (L_n - L_0) / \text{Gauge length}$$

----- (22 - 1)

where:

- L_n is the reading taken at the n^{th} days of storage in sodium hydroxide solution
- L_0 is the measurement of specimen before subjection to sodium hydroxide solution

Gauge length is the distance between inner ends of the metal pins, measured to the nearest 1 mm (i.e. L_i minus the total length of the top and bottom gauge studs).

22.9.2 Expansion recording and further examination

The average expansion of the specimens for a

given period shall be recorded and reported to the nearest 0.01 %. In the case of breakage of specimen during tests, the test will be considered valid provided the average is based on at least four specimens (at least two from each of the two batches). For average values of expansion greater than 0.10 %, the repeatability is considered satisfactory if the expansion of each specimen is within 10 % of the average value. For average values of expansion less than 0.10 %, the repeatability is considered satisfactory if, for each specimen, the deviation from the average value is within 0.01 %. If these values are exceeded, the test shall be deemed invalid and the test shall be repeated.

After the final measurement, the specimens shall be examined and any relevant features recorded. Warping, if observed, shall be measured on 3 moulded surfaces by placing the specimens on a plane surface, with curved ends facing downwards, and measuring the maximum separation between the specimen and the surface to the nearest 0.2 mm. Location, type and pattern of any cracking should also be recorded.

22.9.3 Interpretation of results

The potential alkali-reactivity of aggregate can be assessed from the results established from clause 22.9.1 by using Table 10 below :

Table 10 - Potential Alkali-reactivity of Aggregate for Mortar Bar Test

| Expansion After 14 Days of Immersion in NaOH solution (%) | Potential Reactivity |
|---|----------------------|
| < 0.10 | Non-reactive |
| 0.10 to 0.20 | Potentially reactive |
| > 0.20 | Reactive |

NOTE. *If the mortar-bars exhibit an expansion that is judged to be deleterious, a petrographic examination of the interior of the bars that have expanded most should be carried out together with an examination of the internal crack pattern to confirm that the cause of expansion is likely to be an alkali-silica reaction. If it has been concluded from the expansion results and supplementary examination of the prisms that a given aggregate should be considered potentially alkali-reactive, additional studies using the section 23 of this Section (The Concrete Prism Test), may be appropriate to develop further information on its potential alkali-reactivity and to evaluate the effect of coarse aggregate, different aggregate gradings and different alkali contents of the concrete.*

22.10 TEST REPORT

The report shall affirm that the test was carried out in accordance with this Standard and shall include the following:

- (a) Identification and source of the aggregate sample and reference to petrographic analysis, if available.
- (b) Type and maximum size of the aggregate.
- (c) Type of processing undertaken on the aggregate sample in the laboratory (washing, drying, crushing, sieving etc.).
- (d) Grading of the aggregate as used in the test.
- (e) Identification and source of the Portland cement.
- (f) Alkali content of the cement expressed as equivalent sodium oxide (% Na₂O + 0.658 % K₂O).
- (g) Autoclave expansion or Le Chatelier value of the cement.
- (h) Blaine fineness of the cement.
- (i) Workability (i.e. flow value) of mortar.
- (j) Type and content of superplasticizer (if used to achieve the mortar workability in the

range of 105 % to 120 %), and its contribution to the alkali content of the mortar mix.

- (k) Size of mortar-bars.
- (l) Tested combinations of the aggregate with a reference non-reactive fine material (if the pessimum behaviour has been investigated).
- (m) Initial expansion of the bars after 24 h of storage in water at 80 °C to the nearest 0.002 mm.
- (n) Average percentage length change after each measurement of the specimens to the nearest 0.001 %.
- (o) A graph of the percentage length change against time from the zero reading to the end of the 14-day period of immersion in NaOH solution.
- (p) Results of any warping measurements of the specimens.
- (q) Any significant features revealed by examination of the specimens and the sodium hydroxide solution during and after the test.
- (r) Name and signature of person responsible for the test.

SECTION 23

DETERMINATION OF ALKALI SILICA REACTION POTENTIAL BY CONCRETE PRISM TEST

23.1 SCOPE

The method covers the measurement of expansion of concrete produced by alkali-silica reaction. It enables the effect of specific combinations of aggregates to be investigated.

NOTE. *With some reactive aggregates it has been found that there is a proportion of reactive constituents in the aggregate that leads to maximum expansion. This proportion is called the "pessimum content" and the relationship between expansion and reactive constituents content is called the "pessimum behaviour" of the reactive aggregate.*

23.2 APPARATUS

The following apparatus is required :

- (a) Mixer for laboratory use (see Cl. C1.1).
- (b) Container for concrete prism test (see Cl. C2.4).
- (c) Calliper (see Cl. C7).
- (d) Balance (see Cl. C8.1 & C8.3).
- (e) Test sieves for concrete prism test (see Cl. C23.4).
- (f) Prism moulds for concrete prism test (see Cl. C35.4).
- (g) Length comparator (see Cl. C40).
- (h) Storage cabinet/Temperature controlled cabinet (see Cl. C41.1).
- (i) Temperature control cabinet (see Cl. C41.2).
- (j) Thermometer (see Cl. C42).
- (k) Polythene lay-flat tubing (see Cl. C43).
- (l) Polythene bag (see Cl. C44).
- (m) Twill-weave cotton cloth (see Cl. C45).
- (n) Stop watch (see Cl. C46).

NOTE. *There is contradictory information on the advantages of wrapping or not wrapping specimens and the use of containers containing single or multiple specimens. In order to maximise reproducibility and the chances of maintaining a high and uniform humidity, the wrapping procedure and the use of containers for single specimens is specified in this method. It has been found that wrapping of specimens typically produces higher expansions than equivalent unwrapped specimens.*

Further details of the apparatus are given in Appendix C.

23.3 CASTING AND MEASUREMENT ENVIRONMENT

The casting and measurement of the test specimens shall be carried out in a laboratory with the ambient temperature maintained at (20 ± 2) °C and the relative humidity not less than 60 %.

NOTE. *Cooling to a standard temperature is necessary both to obtain reproducible results and to protect the health of the operator.*

23.4 REAGENTS

23.4.1 Water

Deionised or distilled water.

23.4.2 Sodium hydroxide (NaOH)

Sodium hydroxide (NaOH) shall be at least technical grade.

NOTE. *Reliable safety precautions should be taken and suitable personal protective equipment should always be used to avoid the hazards of the hot alkaline solution such as severe burns and injury to unprotected skin and eyes.*

23.5 MATERIAL

23.5.1 Cement

Portland Cement CEM1 or ASTM Type 1 with known total alkali content of range 0.9 % - 1.2 % sodium oxide equivalent.

NOTE *Suitable reference cements are available from Norcem A.S, R & D Department, 3950 Brevik, Norway, or from National Council for Cement and Building Materials, Calibration Testing & Quality Control, 34 Km Stone, Delhi-Mathura Road (NH-2), Ballabgarh 121 004, Haryana State, India.*

23.5.2 Increasing alkali level of binder

Sodium hydroxide shall be added to the concrete mix water so as to increase the alkali content of the binder to (1.25 ± 0.05) % sodium oxide equivalent.

Sample calculation for determining the amount of NaOH to be added to increase the alkali content from 1 % to 1.25 % shall be as follows :

| | | |
|---|---|---------------------------|
| Cement content of 1 m ³ of concrete | | |
| = | | 440 kg. |
| Amount of alkaline in concrete | | |
| = 440 x 0.01 | = | 4.4 kg. |
| Specified amount of alkali in concrete | | |
| = 440 x 0.0125 | = | 5.5 kg. |
| Amount to be added per m ³ | | |
| = 5.5 - 4.4 | = | 1.1 kg. |
| sodium oxide equivalent | | |
| Conversion factor of sodium oxide to sodium hydroxide: 1.291. | | |
| Amount of sodium hydroxide required | | |
| = 1.1 x 1.291 | = | 1.420 kg/m ³ . |

23.6 PREPARATION OF AGGREGATE SAMPLE

23.6.1 Aggregates combination

The aggregate combination shall consist of one or more of the following:

- (i) the fine and coarse test aggregates;
- (ii) the fine test aggregate combined with a non-reactive coarse aggregate;
- (iii) the coarse test aggregate combined with a non-reactive fine aggregate.

The expansions of non-reactive fine or coarse aggregate in the accelerated mortar-bar test as described in Section 22 shall be less than 0.05 % at 14 days of immersion in sodium hydroxide solution.

NOTE. This expansion limit is deliberately more restrictive than is generally applied to identify non-reactive aggregates to ensure that the non-reactive reference aggregate has minimal effect on the results of this test.

23.6.2 Suggested aggregates proportion

The aggregate fractions shall be combined in

mass proportions on a dry basis of 30 % fine aggregate (4 mm to 0 mm), 30 % coarse aggregate (10 mm to 4 mm) and 40 % coarse aggregate (20 mm to 10 mm).

Where the shape and texture of the aggregate renders a 70/30 ratio of coarse to fine aggregate unworkable, the ratio may be adjusted on an absolute volume basis, but the maximum content of coarse aggregate with which full compaction can be achieved should be employed.

NOTE. If an assessment of the aggregate combination to be used in a specified job is needed, the aggregate shall be combined in the proportions and grading specified.

23.7 CONCRETE MIX DESIGN

23.7.1 Volume proportion of mix

In moulding of specimen, the mix proportion of concrete associated with the aggregate proportions as described in clause 23.6.2 is given in Table 11.

Table 11 - Volume Proportion of Mix

| Material | Proportion by Volume (%) |
|--------------------------------|--------------------------|
| Cement content | 14 |
| Free (effective) water content | 20 |
| Coarse aggregate (20 to 10 mm) | 26 |
| Coarse aggregate (10 to 4 mm) | 20 |
| Fine aggregate (below 4 mm) | 20 |

23.7.2 Weight proportion of mix

Volume proportions in clause 23.7.1 are specified in order to maintain a constant amount of cement (and hence alkali) per unit volume of aggregate when aggregates of different density are tested. In preparation of the quantity of material required in the mix, an example calculation of weight proportion of material per m³ of concrete is given in Table 12.

Table 12 - Weight Proportion of Concrete Mix

| Material | Vol. per m ³ of Concrete (m ³) | Measured Density (kg/m ³) | Weight (kg) |
|--------------------------------|---|---------------------------------------|-------------|
| Cement content | 0.14 | 3200 | 448 |
| Free (effective) water content | 0.20 | 1000 | 200 |
| Coarse aggregate (20 to 10 mm) | 0.26 | 2650 | 689 |
| Coarse aggregate (10 to 4 mm) | 0.20 | 2650 | 530 |
| Fine aggregate (below 4 mm) | 0.20 | 2650 | 530 |

NOTE. The free (or effective) water is the water available for hydration of cement and for the workability of the fresh concrete. The total water added to the mix is the free water plus water absorbed by the aggregate to bring it to a saturated surface dry condition.

23.8 PROCEDURE

23.8.1 Moulding test specimen

23.8.1.1 Mixing

At least three 75 mm x 75 mm x 250 mm specimens shall be made out of each batches of concrete for every aggregate to be tested.

Sodium hydroxide shall be added to the mixing water before mixing and ensuring all is dissolved before use. The aggregates shall be placed in the mixing pan followed by adding half of mixing water containing sodium hydroxide. The materials shall be mixed for 2 minutes and the mixed materials shall then be allowed to stand for 8 minutes with the pan covered to minimize evaporation. The mixer shall be restarted with the cement adding gradually during the next 30 s followed by adding the remainder of the mixing water for the next 30 s and the concrete shall be mixed for further 3 min.

23.8.1.2 Workability of concrete mix

The workability of the concrete mix shall be determined in accordance with Section 2, Part 1 of CS1. It is important that the mix is sufficiently workable to enable good compaction. With some flaky aggregates the above mix might not be sufficiently workable. If the slump of the concrete is less than 80 mm,

a plasticizer (not one combined with an air-entraining agent) should be used. In this case the (small) amount of alkali contributed by the plasticizer should be taken into account in calculating the amount of additional sodium hydroxide to be added.

23.8.1.3 Casting of specimen

The specimen moulds shall be prepared with a suitable releasing agent that will not affect the setting of the cement or leave any residue that will inhibit the penetration of water into the specimen. Two gauge studs shall be fixed in each mould so that they align with the principal axis of the test specimen. Test prism shall be cast from each mix and the concrete shall be compacted into the moulds in two layers of equal depth using mechanical vibration. After completion of vibration, excess concrete shall be removed, the top surface shall be trowelled smooth. The moulded specimens under moist covers shall be placed inside a polythene bag and the end of the bag shall be closed. The bagged prisms shall then be cured at (20 ± 2) °C in relative humidity of not less than 90 % for (24 ± 0.5) hours.

NOTE. It is important to minimize loss of water during this initial hardening stage in order to reduce the risk of shrinkage cracks forming in the specimens.

23.8.2 Storage of specimen after demoulding

After demoulding, one end of each prism shall be designated as the top and this end shall be marked with a number for identification. This end shall be kept uppermost during all subsequent measurements. The prisms shall then be wrapped in wet cloth and polythene bags as described below and shall be stored for 24 hours at (20 ± 2) °C until the initial measurement.

For each prism, a piece of the twill weave cotton cloth 640 mm long and 10 mm wider than the length of the prism shall be cut. Each twill weave cotton cloth shall be saturated with deionised or distilled water (typically 80 ml is required for a cloth 260 mm wide) and one piece shall be wrapped around the four large faces of each prism in two layers. The wrapped prism shall be placed into single pieces of the polythene lay-flat tubing of the same length as the prisms. Each tube shall be smoothed

around the cloth and shall be secured with stout rubber bands.

NOTE. *No wrapping is applied to the end faces of the prisms.*

The wrapped prisms shall be placed in polythene bags with 5 ml of deionised or distilled water pouring over the upper end face of each prism before sealing the bags. Each bagged prism shall then be placed in a storage container, and ensure that deionised or distilled water is contained to at least 20 mm depth.

23.8.3 Measurement

23.8.3.1 Taking zero measurement (L_0)

Twenty-four hours after demoulding, each prism shall be removed from its polythene bag, but leave the wrapping and rubber bands undisturbed. The gauge length (L) of each prism shall be measured to the nearest 1 mm using caliper and the prisms shall be weighed to the nearest 5 g (W_0). The initial length of the prism with stud (L_0) shall be measured using length comparator. These and all subsequent measurements shall be made at a temperature of $(20 \pm 2)^\circ\text{C}$.

Immediately after each measurement, the wrapped prism shall be replaced in its polythene bag with 5 ml of deionised or distilled water pouring over the upper end of the prism before sealing the bag. The container shall be verified that there is at least 20 mm minimum amount of deionised or distilled water in before placing the bagged prisms inside. The lids shall then be replaced, sealed and stored at $(20 \pm 2)^\circ\text{C}$ until the next measurement (L_7 , W_7), which shall be at 7 days after mixing.

NOTE. *The measuring device should be checked with the reference bar prior to and after measurement of each set of specimens. If the difference between the initial and final measurements on the reference bar exceeds 3 μm , all the measurement shall be repeated. The ends of the reference rod, prism studs and the contact surfaces of the comparator shall be cleaned prior to taking a comparator reading. Also, the rod or the prism shall be slowly rotated relative to the measuring device while reading is being taken in order to achieve a consistent result. If the rotation caused a small change in the gauge reading, the minimum reading of the gauge shall be recorded.*

NOTE. *Maintenance of a consistently high humidity within each concrete specimen is critical to obtaining good reproducibility in this method. It is important therefore that the measurement of each prism is carried out without delay, so that the wrapped prisms are outside their sealed*

bags for the shortest possible time, thus avoiding excessive drying from the bare end faces.

23.8.3.2 Subsequent measurement (L_t)

After the 7 day measurement, the containers shall be stored in a cabinet maintaining at $(38 \pm 2)^\circ\text{C}$ until further measurement is required. Twenty four hours before making each set of further measurements, the prisms shall be removed from the cabinet and cooled at $(20 \pm 2)^\circ\text{C}$ whilst remaining inside the containers.

At the end of periods 2, 4, 13, 26, and 52 weeks after mixing, the prisms shall be removed from their containers. The comparator readings on prisms shall be taken and followed by weighing the prisms to the nearest 5 g.

NOTE. *Some types of slowly reacting aggregate may not exhibit deleterious expansion after 52 weeks. Depending on local experience, the length of the test may be extended.*

23.9 CALCULATION AND REPORTING OF RESULTS

23.9.1 Expansion

The increase in length and weight for each prism for each period of measurement from the difference between the initial comparator or weight measurement (L_0 , W_0) and the comparator or weight measurement after that period (L_t , W_t) shall be calculated.

Each increase as a percentage of the initial length or weight of the corresponding prism to the nearest 0.01 % shall be calculated.

For example, at 52 weeks the percentage length change E_{52} of a prism is given by :

$$E_{52} (\%) = 100 \times (L_{52} - L_0)/L \quad \text{-----(23 - 1)}$$

where

L_{52} is the comparator measurement at 52 weeks age

L_0 is the initial comparator measurement of the prism

L is the gauge length of that prism (in mm)

Also the mean length and weight change of the

three prisms for each measurement age shall be calculated to the nearest 0.005 %.

23.9.2 Further Examination

After the final measurement, the prisms shall be examined for any cracking, gel exudations, warping or other features.

23.9.3 Interpretation of results

The potential alkali-reactivity of aggregate can be assessed from the results established from clause 23.9.1 by using Table 13 below:

Table 13 - Potential Alkali-reactivity of Aggregate for Concrete Prism Test

| Expansion After 52 Weeks (%) | Potential Reactivity |
|------------------------------|----------------------|
| < 0.05 | Non-reactive |
| 0.05 to 0.10 | Potentially reactive |
| > 0.10 | Reactive |

NOTE. If the prisms exhibit an average expansion which is judged to be deleterious, a petrographic examination of the interior of the prism which has expanded most should be carried out, together with an examination of the internal crack pattern, to confirm that the cause of the expansion is likely to be an alkali-silica reaction.

23.10 TEST REPORT

The report shall affirm that the test was carried out in accordance with this Standard and shall include the following :

- (a) Identification and source of the aggregate sample and reference to petrographic analysis, if available.
- (b) Identification and source of the Portland cement.
- (c) The mix design of concrete.
- (d) Size of prism used.
- (e) Alkali content of the cement expressed as equivalent sodium oxide (% Na₂O + 0.658 % K₂O) and any alkali additions.
- (f) The slump of concrete.
- (g) Type and content of superplasticizer and its contribution to the alkali content of the concrete mix.
- (h) Tested combinations of the aggregate with a reference non-reactive fine material (if the pessimum behaviour has been investigated).
- (i) Each increase as a percentage of the initial length or weight of the corresponding prism to the nearest 0.01 %.
- (j) The mean length and weight change of the three prisms for each measurement age to the nearest 0.005 %.
- (k) A graph of the percentage length and weight change against time from the zero reading to the end of the test.
- (l) Results of any cracking, gel exudations, warping or other features.
- (m) Name and signature of person responsible for the test.

APPENDIX C

APPARATUS

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APPENDIX C - APPARATUS

C1 Mixer

C1.1 Mixer for laboratory use

The mixer shall be rotating pan or tilting drum type of capacity sufficient to mix batches large enough to provide samples for the tests to be carried out.

C1.2 Mixer for mortar bar test

Mixer, paddle and mixing bowl, as used in the procedure for testing the strength of cement with plastic mortar (e.g. BS EN 196-1:2005 Amd No. 1/2013).

C2 Container

C2.1 Sample container

A bucket or other suitable container made of plastic or metal, of minimum 8-litre capacity.

C2.2 Water container

The water container shall be fitted with a device to maintain the water level constant, and be of sufficient size to allow the specimen on the stirrup to be fully immersed to constant depth.

C2.3 Containers for mortar bar test

Rigid containers for the test specimens shall be made of plastic or other material resistant to corrosion by a solution of sodium hydroxide at a temperature of 80 °C for a prolonged period of time. Each container must be of such dimension and shape to accommodate at least three specimens and must be provided with lids or other suitable means to prevent loss of moisture by leaking or evaporation. The prisms must be positioned and supported in such a way that the solution has access to the whole of the bar. It should further be ensured that the specimens do not touch each other or the sides of the container. The specimens, if stood upright in the solution, shall not be supported by the steel pins.

C2.4 Container for concrete prism test

Container shall be suitable to maintain a high relative humidity around the wrapped prism during storage. A suitable design of the container is shown in Figure 23.

C3 Sample tray

The sample tray shall be at least 900 mm x 900 mm in area, made of metal and have sufficient capacity to contain the concrete sample. It shall have sides to prevent the loss of water or cement paste.

C4 Scoop

The scoop should be made of metal of a size suitable for sampling concrete. The scoop shall not allow water to escape from concrete contained in it.

C5 Compression testing machine

The compression testing machine shall be suited to the size of the specimen and the expected load, and shall comply with BS EN 12390:Part 4 and Clauses C5.1 to C5.5 of this Standard. The performance of the machine shall be verified in accordance with Appendix D of this Standard.

The compression testing machine shall utilize automatic means to record essential test information, including sample identification, machine identification, test date and time, pacer rate and the maximum load. Printed hardcopies or properly controlled and protected computer records containing the above information and the corresponding test results shall be kept.

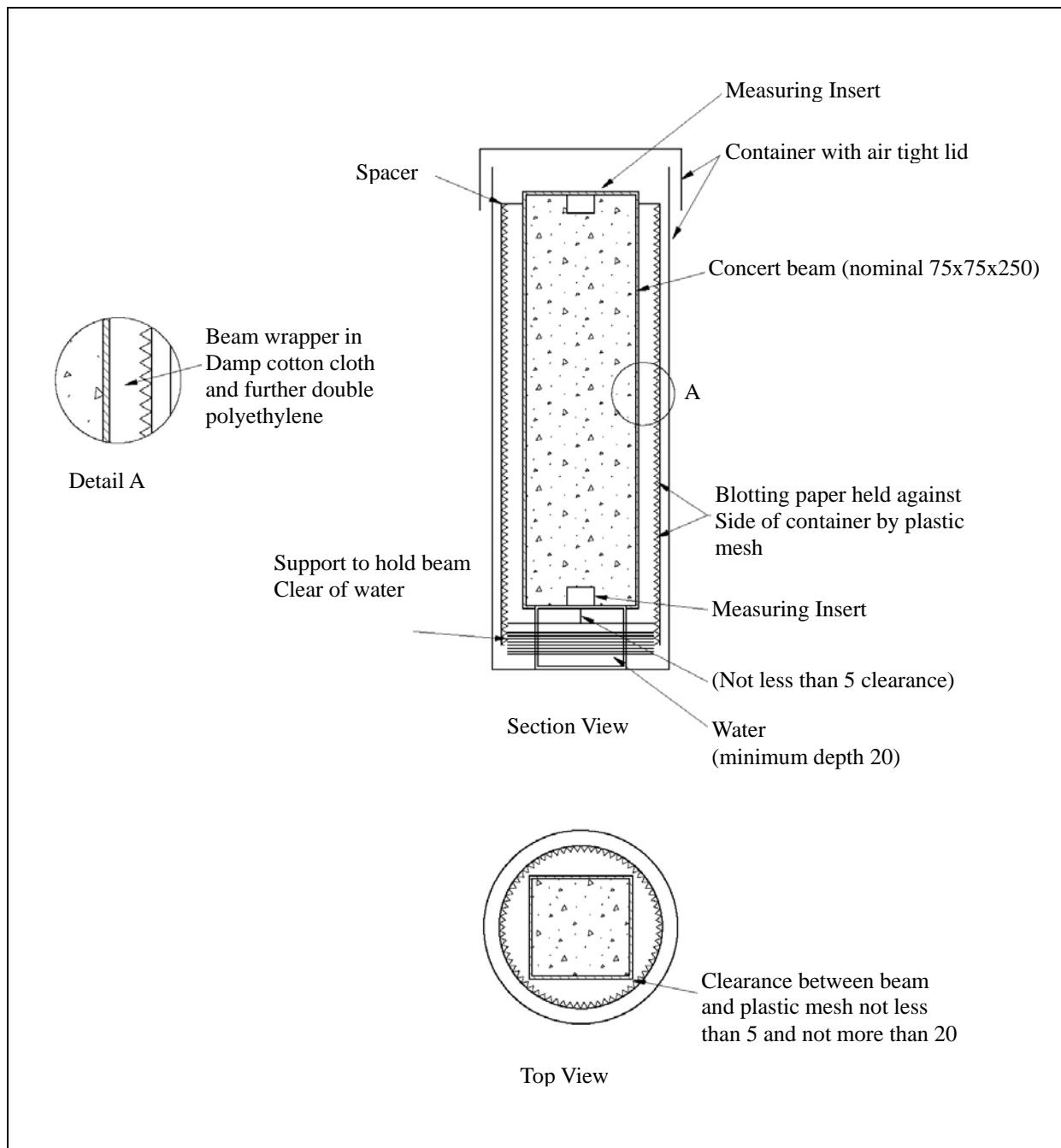


Figure 23 - Container to Provide Humid Environment Around Concrete Prism

C5.1 Loading

C5.1.1 Load control

The machine shall be capable of applying the load at the specified rate, uniformly, without shock, using automatic control.

C5.1.2 Load pacer

The machine shall be equipped with an automatic load control and a load pacer shall be fitted to enable the operator to manipulate the machine controls to maintain the specified rate.

If the pacer is fitted with a variable speed control or has pre-set speeds, then once the variable speed control has been set, or a preset speed has been chosen, the pacer speed shall remain within $\pm 5\%$ of specified speed over the operating range.

C5.1.3 Force indicator

The machine shall be provided with the following:

- (a) An electrical load indicators, with a visual display.
- (b) A resettable device which registers the maximum load sustained by the specimen.

NOTE. *The visual display may be supplemented by recording devices e.g. punched tape or printout recorders, that are calibrated to the same accuracy as the display.*

- (c) The machine load range shall be chosen so that the specimens can be expected to fracture in the part of the range which is certified to be accurate to $\pm 1\%$ or $\pm 2\%$ of the indicated load (i.e. normally the upper four-fifths of the range).

The accuracy of force indication shall be maintained under temperature of $(20 \pm 10)^\circ\text{C}$.

NOTE. *Machines accurate to $\pm 1\%$ are preferred.*

NOTE. *Where electrical or other interference exists this may affect the accuracy of load indication, and special provisions to overcome this interference may be necessary.*

C5.1.4 Means of applying the load

The machine shall apply the load to the specimen either in direct contact with the machine platens or with auxiliary platens interposed between each machine platen and the specimen.

C5.2 Machine platens

In order to avoid excessive platen deformation during loading, the machine platens shall be adequately supported over the area in contact with the specimens.

The platens shall be made of a material which, when tested in accordance with BS EN ISO 6507-1:2005 Amd No. 1/2013, shall have a hardness value of at least 550 HV 30. Also the material shall not deform irreversibly when the machine is used.

The upper platen shall incorporate a ball-seating. The upper platen and the ball-seating may be constructed separately or in one piece.

The flatness tolerance for the area of each platen in contact with the specimen or auxiliary platen shall be $\pm 0.03\text{ mm}$. The contact areas of the platens shall be checked for flatness at annual intervals.

The R_a value for the surface texture of the contact area of each platen shall be between $0.4\text{ }\mu\text{m}$ and $3.2\text{ }\mu\text{m}$ when assessed in accordance with BS 1134:2010 Amd No. 1/2013.

NOTE. *Roughness values of finishes by common manufacturing processes are given in Table 1 of BS 1134:2010 Amd No. 1/2013.*

C5.3 Auxiliary platens

Auxiliary platens may be used in order to save wear on the machine platens and to minimize the effect of overfilled sample on strength measurement. The top auxiliary platen shall rest on and be aligned with the cube. It shall not be fixed to the upper machine platen.

The auxiliary platens shall be made of a material which, when tested in accordance with ISO 6507-1, shall have a hardness value of at least 550 HV 30. Also the material shall not deform irreversibly when the machine is used.

The distance between either pair of opposite edges of a square auxiliary platen, or the diameter of a circular platen, shall be the nominal size of the specimen (i.e. 100 mm or 150 mm) $^{+0.2}_{-0.0}$ mm and thickness of the platen shall be at least 23 mm.

The flatness tolerance for each contact face of the platens shall be ± 0.03 mm. The contact face of the auxiliary platens shall be checked for flatness at annual intervals. If auxiliary platens, worn by direct contact with the test specimens, are resurfaced they shall then be checked for size, parallelism, flatness, surface texture and hardness to ensure compliance with this clause. In case of hardened auxiliary platens are resurfaced they shall be re-hardened and checked for hardness before checking for the dimensional requirements of this case.

The parallelism tolerance for one contact face of the auxiliary platen with respect to the other contact face as datum shall be ± 0.05 mm.

The R_a value for the surface texture of the contact faces of the auxiliary platen shall be between 0.4 μm and 3.2 μm when assessed in accordance with BS 1134:2010 Amd No. 1/2013.

NOTE. Roughness values of finishes produced by common manufacturing processes are given in Table 1 of BS 1134:2010 Amd No. 1/2013.

C5.4 Spacing blocks

If it is required to reduce the distance between the machine platens, up to four spacing blocks may be located beneath the lower machine platen. Spacing blocks shall be either circular or square in section and shall comply with the flatness and parallelism tolerances required for auxiliary platens. Spacing blocks shall be positively located in the horizontal plane and centrally on the vertical machine axis. Spacing blocks shall not be placed in contact with the specimen.

Spacing blocks used beneath the lower machine platen shall be minimum 200 mm in diameter or in length of side if square.

C5.5 Loading of specimen

Provision shall be made for positive and

accurate location in the horizontal plane of the specimen or auxiliary platen on the lower machine platen. The lower machine platen shall be provided with centring lines, location cams or other fixtures for centring specimen or auxiliary platen. If positive physical location is used for position specimens or auxiliary platens, then any locating device shall not restrict the deformation of the specimen during the test. The centring lines, location cams or other fixtures for centring specimen in the platens shall be checked at least once a year. Visual location alone is unacceptable.

C6 Cube checking jig (see Figure 24)

The jig shall be made of steel and consist of a base plate and two vertical side plates all at right angles to each other assembled so that the internal vertical surfaces are $150^{+0.2}_{-0.0}$ mm square. The tolerance for perpendicularity shall be ± 0.2 mm between each pair of faces. The tolerance for flatness of each face shall be ± 0.03 mm. Provided that these tolerances are met it may be convenient to use half a standard cube mould securely fixed to a base plate.

Alternative means of effectively measuring perpendicularity of specimen may be used.

C7 Calliper

The vernier calliper shall be capable of measuring specimen dimensions to an accuracy of 0.1 mm. Other means of measuring specimen dimensions may be used provided the accuracy is 0.1 mm or better.

C8 Weighing equipment

C8.1 Balance

The balance shall be capable of weighing up to 20 kg to an accuracy of 0.1 % of the mass or better.

C8.2 Balance with stirrup

The balance shall be equipped with a stirrup for weighing the specimen in both air and water to an accuracy of 0.1 % of the mass. Some

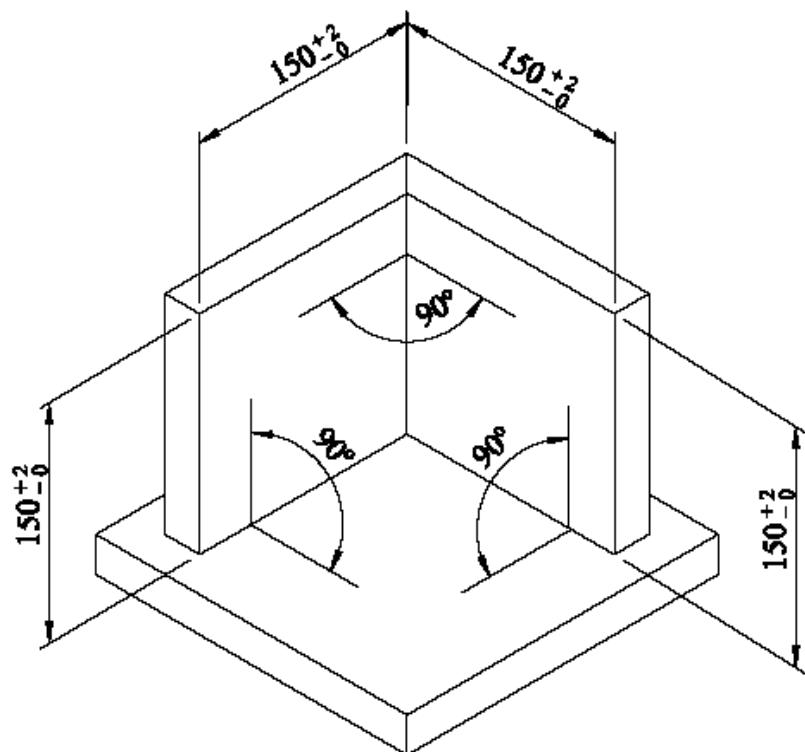
suitable types of stirrup are shown in Figure 25.

C8.3 Balance

Balance shall be minimum capacity of 100 g with an accuracy of 0.1 % of the mass or better.

C9 Feeler gauges

The feeler gauges shall be capable of measuring a gap up to 2 mm.



All dimensions are in millimetres

Figure 24 - Cube Checking Jig

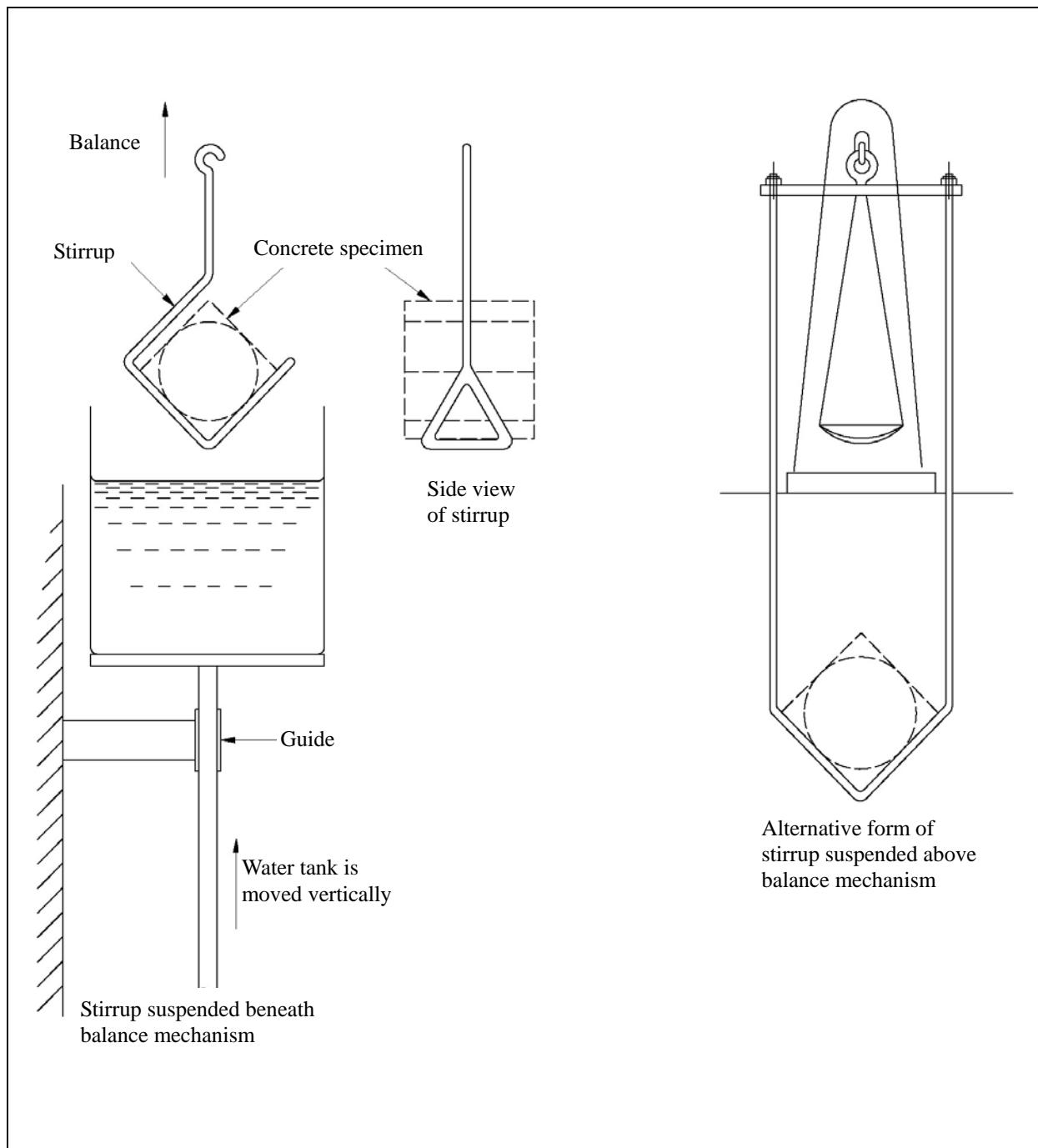


Figure 25 - Typical Stirrup Arrangements for the Determination of the Volume of Concrete by Water Displacement

C10 Jig for tensile splitting strength test

A suitable jig is shown in Figure 14. The jig and/or the machine shall have a device to position the jig correctly on the lower platen, with the specimen central.

C11 Packing strips

The packing strips between the specimen and jig or steel loading pieces shall be of hardboard or other suitable material and shall be used only once.

The length of the packing strips shall be greater than the line of contact of the test specimens. The width of the packing strips shall be (15 ± 2) mm and the thickness shall be (4 ± 1) mm.

C12 Steel loading pieces

A steel loading piece shall be placed between the platen of the machine and the packing strip. This piece shall not be shorter than the specimen. For cylindrical specimens it shall be of rectangular cross-section. This loading piece can be incorporated within the jig.

The flatness, parallelism and surface requirements of the steel loading pieces along the length of the strips shall be within the tolerances for auxiliary platens given in C1. C5.3 of this Appendix.

The steel loading pieces shall be replaced when the dimensions are out of tolerance or they are seriously damaged or pitted along the contact face.

C13 Flexural loading device

The device for applying the loads shall consist of two supporting rollers and two load-applying rollers (see Figure 15). All rollers shall be manufactured from steel and shall have a circular cross-section with a diameter of 20 mm to 40 mm; they shall be at least 10 mm longer than the width of the test specimen and shall be adjusted to the position illustrated in Figure 15 to an accuracy of ± 2.0 mm. All rollers except one shall be capable of rotating about their axes and of being inclined in a plane normal to the longitudinal axis of the test specimen.

NOTE. Although the test method describes a method of loading above the specimen on two inner rollers, the load can be applied by the supporting rollers. Also the upper and lower roller positions relative to each other may be reversed.

C14 Coring machine

The coring machine shall have a diamond drilling bit capable of cutting a core to a straightness tolerance of 3 % of the core diameter.

C15 Crushing and grinding equipment

C15.1 Grinding equipment for concrete cores

The grinding equipment shall be capable of producing a surface to the required tolerances.

C15.2 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. It shall be of suitable size and design capable of crushing aggregate, mortar and etc. to the prescribed size fractions.

C16 Steel plate

A horizontal steel plate with an upper surface having a flatness tolerance of ± 0.03 mm, and a Rockwell (Scale B) Hardness Value of at least 95 when tested in accordance with BS 891:Part 1.

C17 Oven

C17.1 Oven at 105 °C

The oven shall be of sufficient size to enable the specimens to be placed in the oven in the manner specified. The temperature of the oven shall be maintained at (105 ± 5) °C.

C17.2 Oven at 80 °C

An oven shall be of suitable size to accommodate the required number of containers maintained at a temperature of (80 ± 2) °C.

C18 Strain measuring apparatus

The strain measuring apparatus shall have an accuracy of ± 5 micro-strain.

Instruments for measuring strain (for example mirror or dial gauge extensometer, resistance strain gauges, inductance gauges, vibrating wire strain gauges) shall have a gauge length of not less than two-thirds of the width or diameter of the test specimen ($2/3d$).

C19 Water penetration test apparatus

The apparatus for determination of depth of penetration of water shall consist of two screwed on plates tightened up by screw-threaded rod. A packing piece and a sealing ring, which shall be made of rubber or other similar material, shall be placed in such a manner that the water pressure can act on the test area and the pressure applied can be continually indicated.

The apparatus shall be set up as shown in Figure 18.

C20 Vacuum saturation apparatus

The vacuum saturation apparatus shall comprise the following:

- (a) Separatory funnel, or other sealable, bottom-draining container shall have a minimum capacity of 500 mL.
- (b) Beaker (1000 mL or larger) or other container shall be capable of holding concrete specimen(s) and water and of fitting into vacuum desiccator.
- (c) Vacuum desiccator (250 mm inside diameter or larger) shall allow two hose connections through a rubber stopper and sleeve or through a rubber stopper only. Each connection must be equipped with a stopcock.

NOTE. Since vacuum will be drawn over water, a vacuum pump should be protected with a water trap, or pump oil should be changed after each operation.

- (d) Vacuum pump shall be capable of maintaining a pressure of less than 50 mm Hg (6650 Pa).
- (e) Vacuum gauge or manometer shall be accurate to ± 5 mm Hg (± 665 Pa) over range 0 - 100 mm Hg (0 - 13300 Pa) pressure.

C21 Specimen cell sealant

- (a) Specimen-cell sealant shall be capable of sealing concrete to poly (methyl methacrylate), for example, Plexiglas, against water and dilute sodium hydroxide and sodium chloride solutions at temperatures up to 90 °C; examples include RTV silicone rubbers, silicone rubber caulkings, other synthetic rubber, sealants, silicone greases, and rubber gaskets.
- (b) Filter papers No. 2, 90 mm diameter (not required if rubber gasket is used for sealant or if sealant can be applied without overflowing from shim onto mesh).

C22 Voltage cell and data readout apparatus

C22.1 Voltage cell

Applied voltage cell (see Figure. 19) shall be two symmetric poly (methyl methacrylate) chambers, each containing electrically conductive mesh and external connectors. One design in common use is shown in Figure. 19. However, other designs are acceptable, provided that the recommended overall dimensions (including dimensions of the fluid reservoir) are similar as shown in Figure 19.

Temperature measuring device (optional) shall be 0 to 120 °C range and accurate to ± 0.5 °C.

C22.2 Data readout (see Figure 20)

Voltage application and data readout apparatus shall be capable of holding (60 ± 0.1) V dc across applied voltage cell over entire range of currents and of displaying voltage accurate to ± 0.1 V and current to ± 1 mA. Apparatus listed below is a possible system meeting this requirement:

- (a) Digital Voltmeter (DVM) shall be 3 digit, minimum, 0 - 99.9 V range, rated accuracy

- $\pm 0.1\%$.
- (b) Digital Voltmeter (DVM) shall be 4½ digit, 0 - 200 mV range, rated accuracy $\pm 0.1\%$.
 - (c) Shunt resistor shall be 100 mV, 10 A rating, tolerance $\pm 0.1\%$. Alternative, a 0.01 Ω resistor, tolerance $\pm 0.1\%$, may be used, but care must be taken to establish very low resistance connections.
 - (d) Constant voltage power supply shall be 0 – 80 V dc, 0 - 2 A, capable of holding voltage constant at (60 ± 0.1) V over entire range of currents.
 - (e) Cable shall be two conductor, No. 14 (1.6 mm), insulated, 600 V.

C23 Test sieves

C23.1 Test sieves for chemical analysis

If mechanical crushing and grinding is used, sieves with apertures of 150 μm and 1.18 mm will be required. If mechanical crushing and grinding is not used, other test sieves with apertures of 5.0 mm, 2.36 mm and 600 μm will be required.

C23.2 General requirements Amd No. 1/2013

Test sieves of metal wire cloth and perforated plate shall comply with ISO 3310-1:2000 and ISO 3310-2:1999 respectively Amd No. 1/2013 and be complete with the appropriate size of lids and receivers.

C23.3 Test sieves for mortar bar test

Test sieves with supplementary sizes in the series (4.75 mm, 2.36 mm, 1.18 mm, 600 μm , 300 μm and 150 μm) or series (5 mm, 2.5 mm, 1.25 mm, 630 μm , 315 μm and 160 μm) can be used. Amd No. 1/2013

C23.4 Test sieves for concrete prism test

Test sieves shall consist of the following aperture size: 20 mm, 10 mm and 4 mm.

C24 Airtight bottles

The bottle shall be made of glass or plastic and with a capacity of between 100 mL to 500 mL.

C25 Concrete saw

The saw shall be fitted with a diamond or carborundum blade.

C26 Centrifuge

The centrifuge shall be capable of operating at a speed (2500 – 3000) rpm and include sample bottles of capacity (150 – 200) mL with sealed caps.

C27 Transmitted-light microscope

The transmitted-light microscope shall have a magnification of 200 - 400.

C28 Furnace

The furnace or furnaces shall be capable of maintaining temperatures of (925 ± 25) °C and (1200 ± 50) °C.

C29 Desiccator

The desiccator shall contain dried magnesium perchlorate.

C30 Vacuum desiccator

The vacuum desiccator shall obtain dried magnesium perchlorate and be capable of maintaining a pressure of less than 13.5 kPa.

C31 Water pump or vacuum pump

The pump shall be capable of reducing the pressure in the vacuum desiccator to less than 13.5 kPa (100 mmHg).

C32 Apparatus for the determination of combined water content

The apparatus shall consist of a small absorption vessel, containing dried magnesium perchlorate, through which air, nitrogen or other suitable gas is passed before entering a silica combustion tube that is heated in furnace capable of reaching 1000 °C, bright red heat. The exit from the combustion tube shall be packed with silver wool to remove any hydrogen sulphide evolved. If silicon 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. A typical apparatus is shown in Figure 26.

C33 Rotary lap-plate

The lap-plate shall use a non-aqueous polishing medium and diamond pastes.

C34 Reflected-light microscope

A reflected-light microscope using magnifications up to x 800.

C35 Mould

C35.1 Cylindrical mould

The mould shall be at least 25 mm in diameter, e.g. a plastic tube.

C35.2 Mould for flow test

Mould shall be a frustum of a cone with height 50 mm, base diameter 100 mm and top diameter 70 mm (e.g. conforming to BS 4551-1:1998 Amd No. 1/2013).

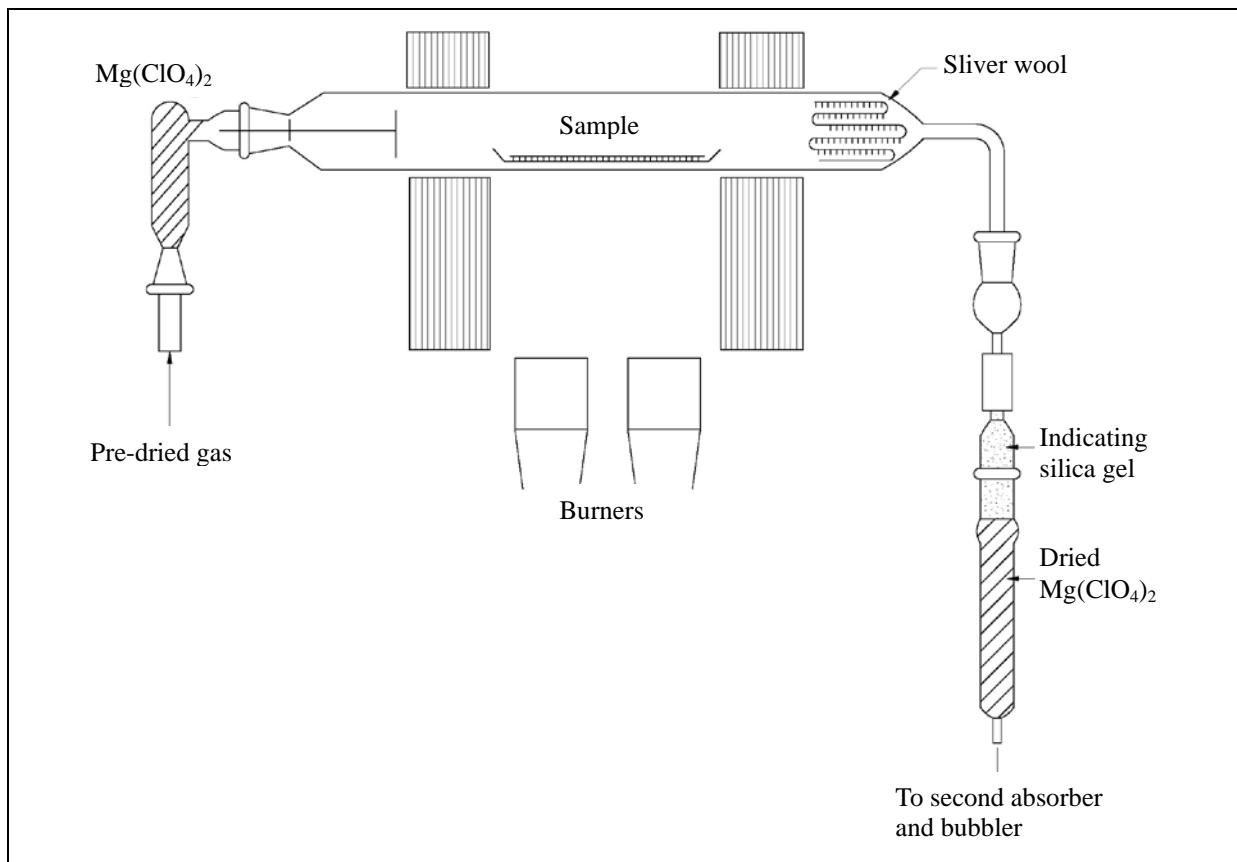


Figure 26 - Apparatus for the Determination of Water Content

C35.3 Prism moulds for mortar bar test

Moulds shall be provided for prisms with a nominal length of 285 mm and a cross section of 25 mm x 25 mm (lengths in the range of 250 mm to 300 mm are also acceptable). The end plates of the moulds must have threaded holes in the centres to take stainless steel or other corrosion resistant metal of suitable hardness hemispherical studs of 6 mm diameter and 20 mm length used for length measurements. All parts of the mould shall be clearly marked with a reference number to enable the mould to be correctly reassembled after demoulding.

C35.4 Prism moulds for concrete prism test

Moulds shall be suitable for casting concrete prism of length (250 ± 50) mm and cross-section (75 ± 5) mm. The moulds shall have the facility for casting stainless steel reference studs into the midpoints of the end faces of the prisms. The end plates of the moulds must have threaded holes in the centres to take stainless steel or other corrosion resistant metal of suitable hardness hemispherical studs of 6 mm diameter and 20 mm length used for length measurements. All parts of the mould shall be clearly marked with a reference number to enable the mould to be correctly reassembled after demoulding.

C36 Apparatus for the determination of carbon dioxide (Method 1)

The apparatus shall consist 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 shall pass 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 27.

C37 Apparatus for the determination of carbon dioxide (Method 2)

The apparatus shall consist of the following :

- A graduated burette of 200 mL – 250 mL capacity and reading to 0.5 mL or better.
- A plastic measuring cylinder of 20 mL capacity.
- A levelling tube which can be raised and lowered on a mounting panel.
- A thermometer of range 0 – 40 °C and accurate to 0.2 °C.
- A barometer.

The apparatus shall be set up as shown in Figure 28.

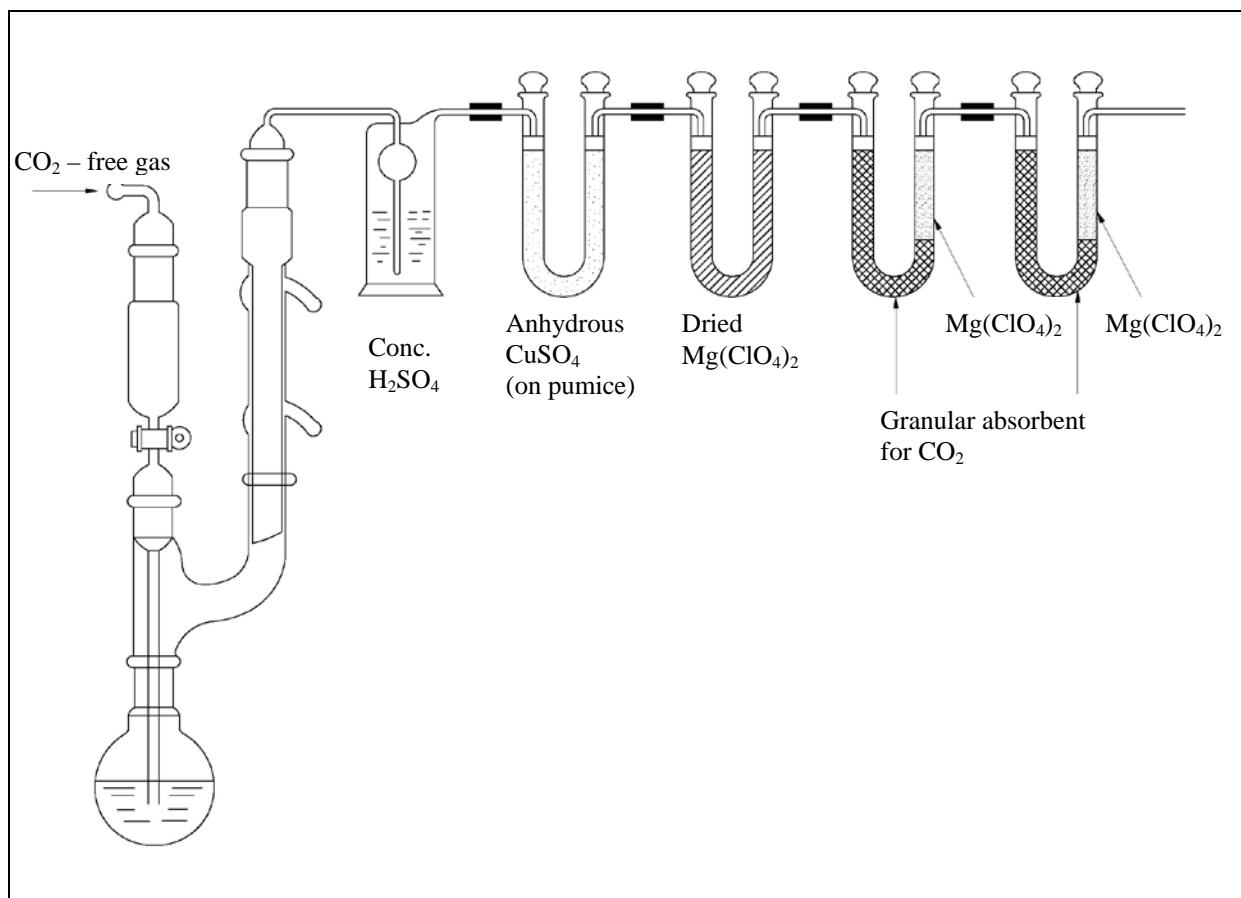


Figure 27 - Apparatus for the Determination of Carbon Dioxide (Method 1)

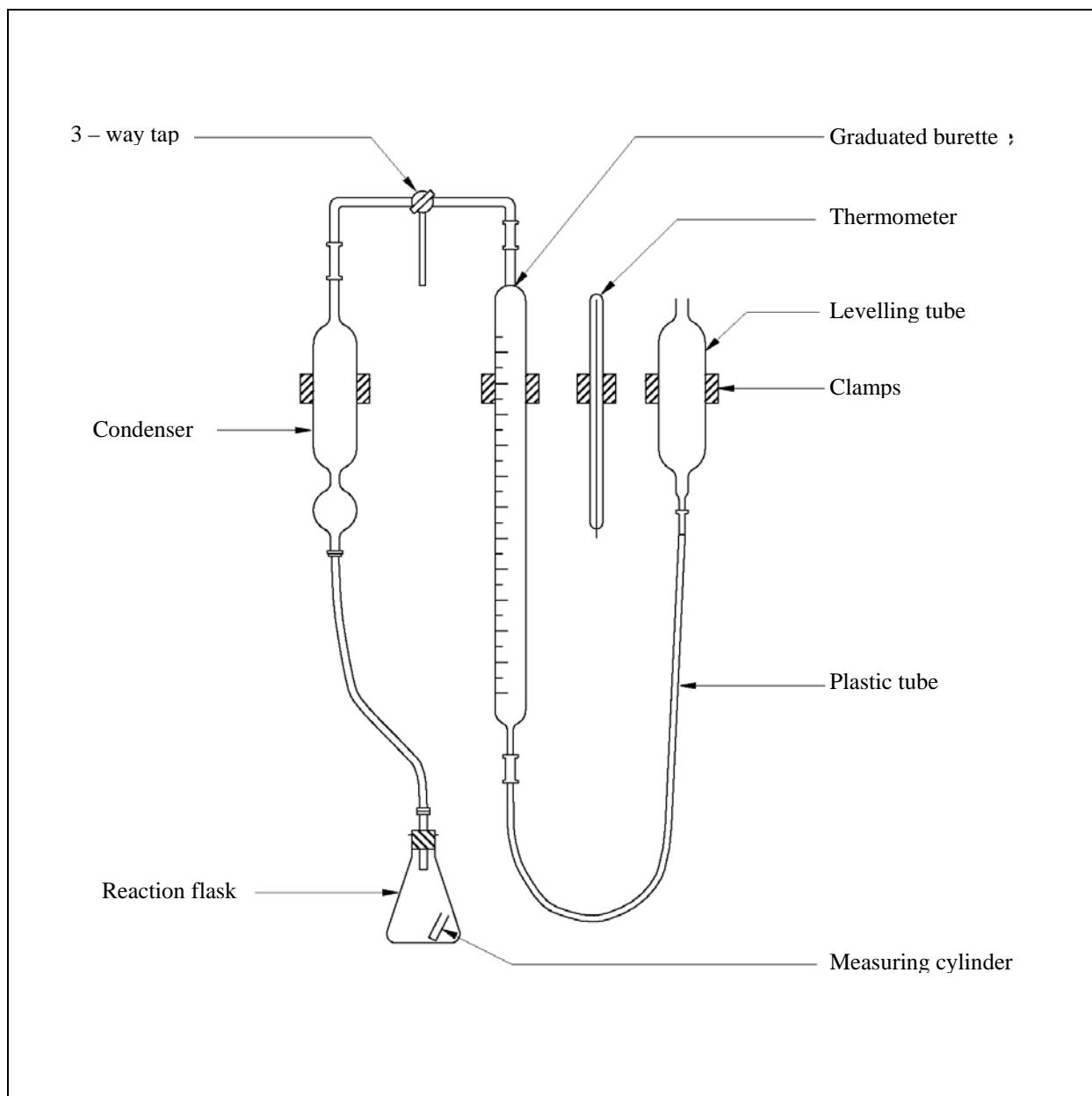


Figure 28 - Apparatus for the Determination of Carbon Dioxide (Method 2)

C38 Flow table

Flow table shall be consisted essentially of a circular rigid table top, with a diameter of about 250 mm, that can be raised vertically by means of a cam, and dropped through a nominal height of 12.7 mm (e.g. conforming to BS 4551-1:1998 Amd No. 1/2013).

C39 Tamper

Tamper shall be made of non-absorbent, non-abrasive, non-brittle material and with a

cross-section of approximately 13 mm x 25 mm. The tamping face shall be flat and at right angles to the length of the tamper.

C40 Length comparator

Length comparator shall be consisted of the following features :

- An apparatus to measure the length of the specimens conveniently and rapidly. The arrangement of the comparator and the range of displacement of the gauge shall be such that specimens can be inserted and removed

- without damage or undue pressure to the beams, reference studs or measuring device. Each stainless steel or other corrosion resistant metal of suitable hardness stud locator of the comparator shall have a hemisphere to accommodate the shape of the reference studs in the beam or reference bar.
- (b) A high-grade dial micrometer, or other measuring device, graduated to read in $2\mu\text{m}$ units and the error throughout the range of traverse shall be no more than 0.005 mm and it shall be mounted rigidly in a vertical orientation. The measuring range shall allow for small variations ($\pm 10\text{ mm}$) from the nominal gauge length of the specimens.
- (c) An Invar reference bar (or similar) of the same nominal length as the specimens for checking the measuring device, before and after each set of readings. Its ends shall be machined to match the shape of the reference studs in the prism.

NOTE. *The gauge length of the rod will be slowly diminished with time as the wear of contact surfaces. It is therefore recommended that a second similar rod be stored for use as a primary standard for periodically checking the regular rod, or for use as spare if necessary.*

An example of a measuring frame is shown in Figure 29.

C41 Temperature control chamber/cabinet

C41.1 Storage cabinet/Temperature controlled cabinet

A storage cabinet or moist chamber shall be maintained at a temperature of $(20 \pm 1 \text{ Amd No. } 1/2013)\text{ }^{\circ}\text{C}$ and a relative humidity not less than 90% (e.g. conforming to BS EN 196-1:2005 Amd No. 1/2013).

C41.2 Temperature control cabinet

Temperature controlled chamber or cabinet shall be capable of maintaining at a temperature of $(38 \pm 2)\text{ }^{\circ}\text{C}$ throughout its storage space.

C42 Thermometer

The thermometer shall be suitable for measuring temperature within the intended range to an accuracy of $0.5\text{ }^{\circ}\text{C}$.

C43 Polythene lay-flat tubing

Polythene lay-flat tubing shall be 180 mm wide, with approximately the same length as the prism.

C44 Polyethylene bag

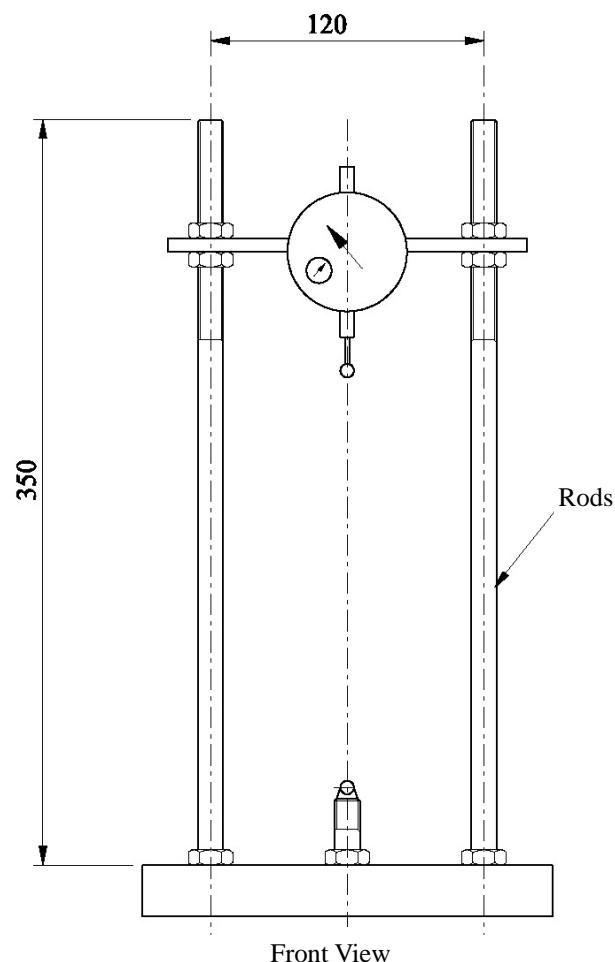
Polyethylene bag shall be approximately $500\text{ mm} \times 250\text{ mm}$.

C45 Twill-weave cotton cloth

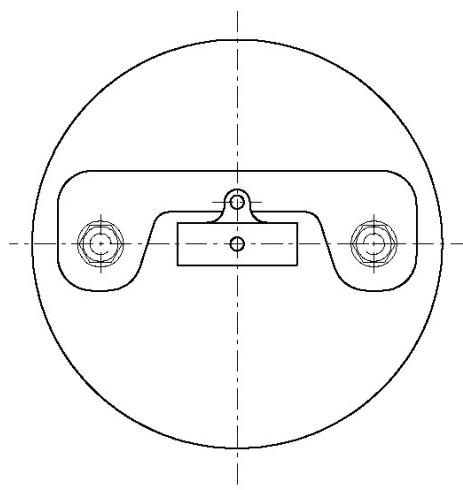
Twill-weave cotton cloth shall be $(240 \pm 30)\text{ g/m}^2$ and at least 10 mm wider than the length of the beam.

C46 Stop watch

The stop-watch or stop-clock shall be accurate to 0.5 second .



Front View



Plan

Figure 29 – Length Comparator

APPENDIX D
**VERIFICATION OF PERFORMANCE OF
COMPRESSION TESTING MACHINE**

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APPENDIX D - VERIFICATION OF PERFORMANCE OF COMPRESSION TESTING MACHINE

D1 Load verification

The machine scales or digital displays shall be verified at intervals of not more than six months, when a machine is moved to a new location or is subject to disturbance, major repairs or adjustments. The verification shall be in accordance with BS EN 12390:Part 4 to ensure compliance with C1. C5 of this Standard.

D2 Performance

When testing concrete cubes or vertical cylinders in compression:

- (a) the component parts of the machine shall be aligned accurately;
- (b) the upper machine platen shall align freely with the upper face of a correctly located specimen or upper auxiliary platen as initial contact is made; and
- (c) the upper machine platen shall then be restrained from tilting with respect to the lower machine platen during loading.

Compliance with these requirements shall be checked on initial commissioning, after subsequent relocation or disturbance, and at intervals of not more than six months by meeting the compliance requirements of C1. D3.3, D3.4 and D3.6. The method shall be in accordance with BS EN 12390:Part 4.

NOTE. Where it can be demonstrated that other devices and methods of verification will provide comparable verification of the requirements, the use of these alternatives is permitted.

D3 Strain gauged column and proving procedure

D3.1 The strain gauged column

The strain gauged column shall be a cylinder of steel of the hardness value of 370 HV 30. It shall be (100 ± 1) mm in diameter and (200 ± 1) mm high. The flatness tolerance for the ends shall be 0.03 mm but the surfaces shall not be convex. The parallelism tolerance shall be 0.06 mm. The perpendicularity tolerance of the cylinder with respect to one end as datum face

shall be 0.03 mm. The roundness tolerance of the ends of the cylinder shall be 0.02 mm, and the whole cylinder shall be within a cylindricity tolerance of 0.04 mm. Centre holes of maximum size 15 mm diameter by 15 mm deep are permitted in the ends of the cylinder.

The column shall be gauged using matched temperature-compensated electrical resistance strain gauges. Four complete bridges, each centred at one of the ends of a pair of orthogonal diameters half way up the cylinder shall be used. Each bridge shall consist of two elements measuring axial strain and two measuring circumferential strain as shown in Figure 30. Each bridge shall be electrically and thermally balanced.

The column shall be supported in a carrying box by circumferential shoulders near the ends of the cylinder. The edge of each shoulder nearest the centre of the cylinder shall be no further than 15 mm from the nearest end of the cylinder. Vertical lines shall be inscribed on the cylinder walls so that they are visible outside the carrying case to indicate the position of the centre lines of the bridges. These lines shall not extend further than 20 mm from the lower end of the cylinder.

The column shall be used with a switch and balance unit which enables the outputs of each of the four bridges to be balanced in the unloaded condition and the bridge outputs to be selected thereafter by operation of a switch.

NOTE. Alternatively, simultaneous display of the four bridge outputs may be used if means are provided to enable the sensitivity of the four channels to be checked and, if necessary, equalized immediately prior to the taking of a series of readings.

The strain gauged column shall be used in conjunction with dedicated strain measuring equipment.

The maximum limit of error for the strain-measuring equipment shall be $\pm 0.1\%$ or five microstrain, whichever is greater.

The strain gauged column with its dedicated strain measuring equipment shall be calibrated to national standards at least every two years.

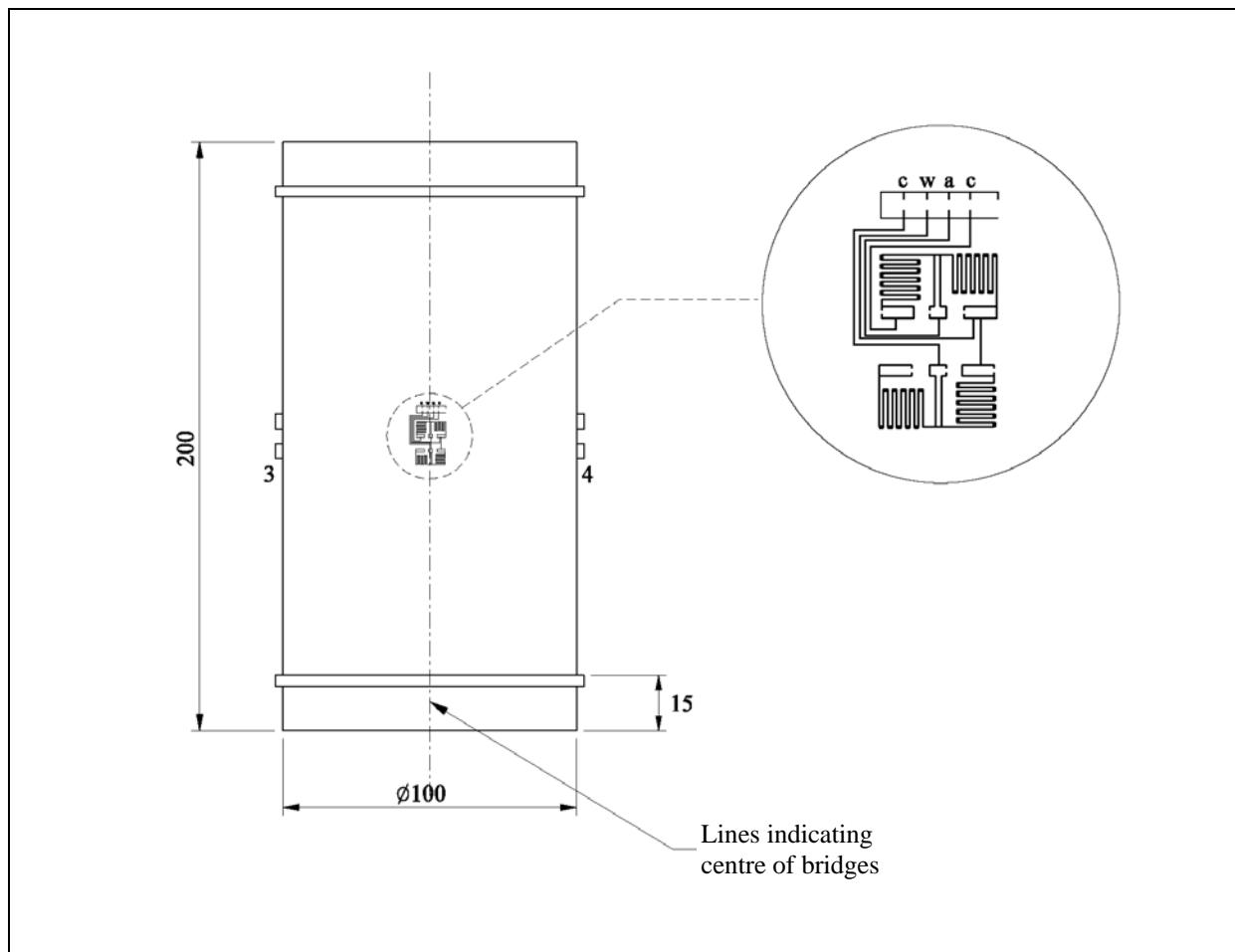


Figure 30 - Gauging of Proving Device

D3.2 Procedure for verifying the self-alignment of the upper machine platen and the component parts of the machine

A 150 mm square auxiliary platen which complies with C1. C5.3 and is not convex shall be located on the lower machine platen or spacing block.

The proving device shall be placed centrally on the square auxiliary platen. The mid-points of the edges of the auxiliary platen shall be designated by A, B, C and D and the four bridge positions on the device designated by 1, 2, 3 and 4. The device shall be positioned by eye as shown in Figure 31.

The distances from the centre of each top edge of the lower auxiliary platen to the nearest point on the bottom edge of the device shall be measured and the position of the device adjusted until the differences between the pairs of measurements from opposite edges of the platen to the device do not exceed 0.1 mm.

NOTE. This may be conveniently achieved by fitting a stop to the edge of the platen and providing accurately machined spacers to centralize the device.

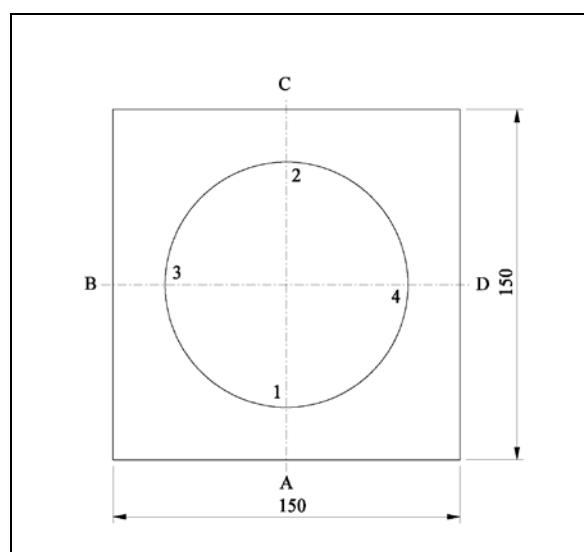


Figure 31 - Positioning of proving device on auxiliary platen

The upper machine platen shall be brought to no closer than 5 mm to the top of the device. It shall then be tilted towards A about axis BD either to its fullest extent or until it touches the device. The upper machine platen shall be gently released and the machine operated so that the upper machine platen aligns with the device. The indicated load on the device shall be increased slowly up to a nominal value of at least 200 kN. The load shall be held constant and the outputs of the four bridges recorded. If the load exceeds 200 kN but does not exceed 220 kN before it can be held constant, the load shall not be reduced before taking the readings. If the load exceeds 220 kN the test shall be restarted.

Using the mean e_m of the four bridge outputs e_1 , e_2 , e_3 and e_4 , the strain ratio $(e_n - e_m)/e_m$ for each bridge shall be calculated, where e_n is the strain at the bridge position under consideration.

The test shall be repeated first with the upper machine platen tilted down towards C about an axis BD, secondly with the upper machine platen tilted down towards B about an axis AC and thirdly with the upper machine platen tilted down towards D about an axis AC.

If the device is correctly machined and gauged, the sensitivity of the four bridges will be equal. However, if this is in doubt, the readings shall be repeated first with bridge 1 adjacent to B, then with bridge 1 adjacent to C and finally with bridge 1 adjacent to D (see Figure 30).

The readings so obtained, together with those obtained with bridge 1 opposite A should be averaged to eliminate differences in bridge sensitivity on the device. This should be done for the readings on all four bridges.

D3.3 Self-alignment of the upper machine platen

The strain ratios at 200 kN for the four different directions of initial platen tilt shall be obtained and compared. The difference between the highest and lowest values for any bridge shall not exceed 0.10.

D3.4 Alignment of the component parts of the machine

If the self-alignment is correct (see C1, D3.3), the mean strain ratios for each of the four bridges shall be calculated. For each bridge, this value shall lie within the range ± 0.10 .

D3.5 Procedure for verifying restraint on movement of the upper platen

If the self-alignment and alignment are correct (see C1, D3.3 and D3.4) the device shall be displaced by (6 ± 0.05) mm from the central position along AC towards A. Without further adjustment of the upper machine platen, the machine shall be operated to bring it into contact with the device and the load applied smoothly. The outputs of the four bridges at nominal loads of 200 kN and 2000 kN shall be recorded. If the machine capacity is less than 2000 kN, the readings at 200 kN and at maximum capacity shall be taken. Care shall be taken to ensure that the output of each of the four bridges is read while the load is held constant. If either nominal load is exceeded by not more than 10 % before it can be held constant, the load shall not be reduced and the readings shall be taken. If either nominal load is exceeded by more than 10 %, the test shall be restarted.

These readings shall be repeated with the device displaced (6 ± 0.05) mm from the central position, firstly along AC towards C, secondly along BD towards B and thirdly along BD towards D. The change in strain ratio per mm offset for displacement along AC and for displacement along BD for each load shall be calculated. If r_1 , r_2 , r_3 and r_4 represents strain ratios for bridges at positions A, C, B and D and a , c , b and d denotes ratio for displacement towards A, C, B and D respectively then for each load the value for displacement along AC shall be calculated as:

$$\frac{(r_{1c} - r_{2c}) - (r_{1a} - r_{2a})}{24} \quad \text{-----(D - 1)}$$

and for displacement along BD as :

$$\frac{(r_{3d} - r_{4d}) - (r_{3b} - r_{4b})}{24} \quad \text{-----(D - 2)}$$

D3.6 Restraint on tilt of the upper platen

The change in strain ratio per mm offset in each of the two directions shall not exceed 0.06 at 200 kN or 0.04 at 2000 kN or at the maximum capacity of the machine if this is less than 2000 kN.

D3.7 Safety requirements

When using the device, particularly when it is at an eccentric setting, care shall be taken to

ensure that the indicated force does not exceed the specified value of the device.

The device shall be clearly marked indicating the '*maximum permitted force when applied centrally*'.

NOTE. *If the device is overloaded in an eccentric position, there is a danger that the horizontal motive forces resulting from a one-side compression of the device can exceed the retaining friction forces between the compression surfaces of the testing machine and those of the device, which would cause the device to be projected violently from the testing zone.*