INDUSTRY/FRD COLLABORATIVE RESEARCH PROGRAMME: ACHIEVING DURABLE AND ECONOMIC CONCRETE CONSTRUCTION IN THE SOUTH AFRICAN CONTEXT

SERIES OF RESEARCH MONOGRAPHS

The work reported in this monograph, and others in the series, has arisen chiefly from a research programme into how to achieve durable and economic concrete construction in the South African context. The present programme has been in operation since 1997, and continues the good work achieved in a previous programme under the Foundation for Research Development. The programme is a joint collaborative effort between research students and staff at the Universities of Cape Town and the Witwatersrand. The work of the research students, in particular, is acknowledged. The monographs are essentially compilations of research papers and reports that have emanated from the programme over the past several years.

The programme has been generously supported financially by a number of industrial sponsors and funding agencies. The authors would like to acknowledge specifically the following:

- The Cement and Concrete Institute, through which cement industry funds have been channelled, and in particular the untiring efforts of Dr Graham Grieve, the C&CI Executive Director, who for many years has chaired the Advisory Panel which oversees the work of the programme;
- Pretoria Portland Cement (PPC), Alpha Cement, Lafarge South Africa (previously Blue Circle), and Natal Portland Cement (NPC), all of which have provided financial assistance and, on occasions, contract sponsorship;
- The Foundation for Research Development (FRD), which has provided bursary assistance for students as well as other financial support, and which has provided administrative and logistic support to the programme;
- LTA Construction, for providing steady financial support;
- The THRIP Programme of the FRD and the South African Department of Trade and Industry, for additional financial support;
- Eskom, which more recently provided financial support through the TESP Programme.

M G Alexander, (UCT); Y Ballim (Wits), J R Mackechnie (UCT), March 1999

See inside back cover for a list of monographs in this series

---

RESEARCH MONOGRAPH NO. 4

Concrete durability index testing manual

M G Alexander
Department of Civil Engineering, University of Cape Town

Y Ballim
Department of Civil Engineering, University of the Witwatersrand

J R Mackechnie
Department of Civil Engineering, University of Cape Town

DEPARTMENTS OF CIVIL ENGINEERING
University of Cape Town
University of the Witwatersrand

Published by the Department of Civil Engineering
University of Cape Town

1999

Printed by UDMS, Observatory, Cape Town

TABLE OF CONTENTS

Acknowledgements *4*

Introduction **5**

1. Method statement for the oxygen permeability test for concrete **8**

2. Method statement for the water soptivity test for concrete **17**

3. Method statement for the chloride conductivity test for concrete **26**

ACKNOWLEDGEMENTS

The authors acknowledge the financial support of the South African cement industry, and of LTA, Eskom, and The Foundation for Research Development. The contribution of Dr Piet Streicher, who did most of the development work for the chloride conductivity test, is also especially recognised, as is the work of the other students and laboratory technicians who have made useful contributions to the development of the test methods given in this monograph.

INTRODUCTION

This manual is intended to provide instructions and guidelines on conducting the three durability index tests for concrete: the oxygen permeability test, the water sorptivity test, and the chloride conductivity test. These tests have been developed over several years as part of work undertaken under a collaborative research programme aimed at improving the durability of concrete construction in the South African context. The programme has been run jointly at the Universities of Cape Town and the Witwatersrand, and has enjoyed strong support of industry and the FRD. Most of the work represents research undertaken by Masters and Doctoral students, and their contributions are gratefully acknowledged.

Ballim, in his PhD research at the University of the Witwatersrand,[1] developed the oxygen permeability test from a previous test proposed by Blight[2] for testing the permeability of asphalt layers. Ballim also developed the water sorptivity test as part of his PhD research. Subsequently, Streicher, at the University of Cape Town, developed the chloride conductivity test in his PhD research.[3] Numerous other students have since applied the index approach to various aspects of the achievement of durable concrete construction. In particular, Mackechnie[4] at UCT was able to show that the index tests could be used both to *characterise* the quality of concrete at an early age, and subsequently to *predict* the performance of concrete in a marine environment.

It is important that the philosophy of index testing be understood. The concrete properties of oxygen permeability, water sorptivity, and chloride conductivity are not measured for their own intrinsic value, but rather to provide *indexes* of concrete quality, in particular the quality of the cover layer of concrete, or the outer surface layer of

---

[1] Ballim, Y, Curing and the durability of concrete, PhD Thesis, University of the Witwatersrand, 1994.

[2] Blight, GE, A falling head permeameter for testing asphalt. Trans. S A Inst. of Civ. Eng., June 1977, 123-126.

[3] Streicher, PE, The development of a rapid chloride test for concrete and its use in engineering practice. PhD Thesis, University of Cape Town, 1997.

[4] Mackechnie, JR, Predictions of reinforced concrete durability in the marine environment. PhD Thesis, University of Cape Town, 1996.

concrete. In this regard, the tests have been shown to be sensitive to the important material, constructional, and environmental factors that influence durability. Thus, the tests provide reproducible engineering measures of the microstructure of the concrete, including the influence of different binder types where the chemistry might play an important role. The tests characterise the quality of concrete as affected by choice of materials and mix proportions, placing, compaction and curing techniques, and environment. They can therefore be used to assist in controlling the quality of concrete at the construction site since the sample retrieval and test techniques can be applied on site. Site experience[5] has shown that it is practicable to apply the index approach on construction sites, and that the index tests are able to quantify the quality of concrete construction.

Several years of experience of using the index tests in our laboratories indicate that the different tests are best suited to assessing the following aspects of concrete quality:

Oxygen permeability test. This test assesses the overall microstructure and macrostructure of the outer surface of cast concrete, and is particularly sensitive to macro-voids and cracks, which act as "short-circuits" for the permeating gas. Thus the test is very useful to assess the state of compaction, presence of bleed voids and channels, and the degree of interconnectedness of the pore structure.

Water sorptivity test. This test measures the physical properties of a generally thin (5-15 mm) layer of surface concrete, by means of the mechanism of capillary suction. It has been found to be very sensitive to the nature and extent of early curing of the cover concrete, and can thus be used on site to assess construction quality. The test is simple to carry out, but it is very important to pre-condition the samples correctly so as to have a reproducible moisture condition at the start of a test.

Chloride conductivity test. This test is specifically intended to assess the resistance of concrete to ingress by chloride ions, such as would occur in marine environments (or in de-icing salt environments). It is very sensitive to binder type and chemistry, and also to the period of pre-saturation with chloride solution. It is intended not so much as a quality

control test on site (although it may be used to check on quality of the mix materials, particularly the binder), but as a test to allow proper material selection and mix optimisation prior to construction.

It must be stressed that caution is required in the proper interpretation of the results of the tests. Extraneous influences such as alteration of the concrete surface by, for example, weathering or salt deposition can markedly affect the results. It should also be appreciated that statistical scatter may be inherently higher than is the norm for concrete strength testing. Nevertheless, care and attention to testing detail can result in acceptably low experimental scatter. Finally, the tests should not be applied outside their intended areas of use, which are primarily as indicators of early-age quality of concrete construction, and as predictors of concrete performance. Applying the tests to aged and altered concretes, for example, can yield results that are difficult to interpret and may be relatively meaningless.

It is strongly recommended that the other monographs in this series be studied in order to more fully understand the application of the index approach to achieving concrete durability. See the inside back cover for a list of the monographs.

It is hoped that the publication of this manual of test methods will encourage other laboratories to begin experimenting with the tests. At the same time, the need to incorporate the approach in re-drafted national specifications is urgent. A database of index test results from around the country will assist in providing criteria for suitable specifications. A national effort is needed to ensure that this process is carried forward.

The equipment described in the test methods that follow can be constructed in the workshops of the Departments of Civil Engineering of the Universities of Cape Town and the Witwatersrand.

---

[5] Bouwer, SM, Practical implementation of index tests for assessment and control of potential concrete durability. MEng Thesis, University of Stellenbosch, 1998.

I. METHOD STATEMENT FOR THE OXYGEN PERMEABILITY TEST FOR CONCRETE

Scope

The oxygen permeability test involves determining the oxygen permeability index of a concrete sample from the rate of pressure decay through the sample when placed in a falling head permeameter. The test gives an indication of the gas permeation properties of concrete.

Referenced document

Ballim, Y (1991). A low cost falling head permeameter for measuring concrete gas permeability. Concrete Beton, No. 61, pp. 13-18.

Summary of test method

The test method consists of measuring the pressure decay of oxygen passed through a 25 mm thick slice of a 68 mm diameter core of concrete placed in a falling head permeameter as shown in Figure 1.1.

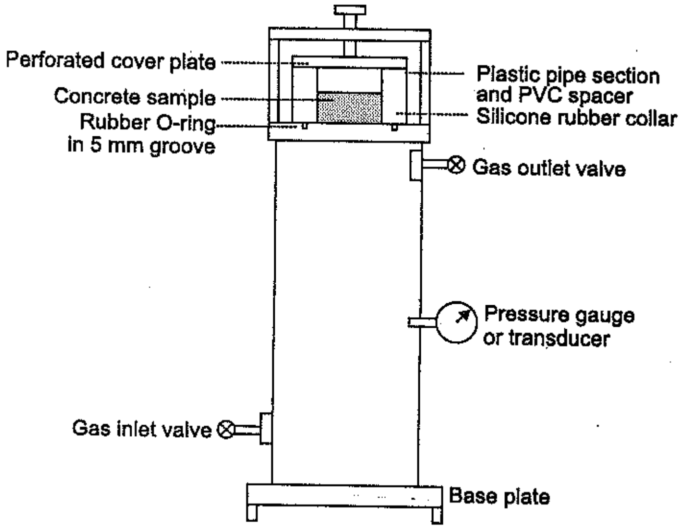

Figure 1.1: Oxygen permeability cell

In the test, oven-dried concrete samples are subjected initially to oxygen at a pressure of 100 kPa and the pressure decay with time is monitored.

The coefficient of permeability is determined from the slope of the line produced when the natural log of the ratio of initial pressure to pressure at any time is plotted against time. The oxygen permeability index is defined as the negative log of the coefficient of permeability. The test can be automated by using pressure transducers linked to a computer-based data logger. One test result is calculated from the mean of three, or preferably four test determinations on the same material. It is recommended that at least one extra sample is prepared for the purpose of re-testing, in the case of a test failing.

Significance and use

a) This test method is suitable for evaluation of materials and mix proportions for design purposes as well as for research and development purposes.

b) This test method is suitable for use as a quality control test on construction projects, provided suitable statistical criteria are used to make judgements.

c) The details of the test method apply to 25 mm thick × 68 mm diameter specimens, (normally taken from the cover layer of concrete). Other specimen thicknesses from 5 mm (mortar) to 40 mm may be used, but different conditioning times may be required.

d) Sample age may have a significant effect on the test results, depending on the type of concrete and the curing procedure. Most concretes, if properly cured, become progressively and significantly less permeable with time.

e) Care should be taken in interpreting the results of this test when it is used on surface-treated concretes, or on concrete surfaces that have been exposed to environmental influences such as carbonation or exposure to marine salts.

Apparatus

a) Oven capable of maintaining a temperature of 50 ± 2°C and a relative humidity below 20 %.

b) Permeability test arrangement. While it is possible to use only one or two cells with manual pressure gauges, for rapid production of results it has been found preferable to have an arrangement as shown in Figure 1.2.

- Permeability cell. The permeability cell is manufactured using a length of welded steel pipe (I.D. = 106 mm, wall thickness = 4 mm) with a volume of approximately 5 *l*. All valve and pressure gauge seals and welds should be air tight at a pressure of 120 kPa. The volume of the steel pipe section should be determined either by dimensional measurement or by measuring the volume of water required to fill the cylinder. The volume of the opening in the top plate as well as the rubber collar annulus below the sample should be included in the assessment of the volume of the cylinder. This volume should be recorded to the nearest 0.01 *l*. The air-tightness of the cell can be tested using a blank sample in place of a concrete sample and by painting all connections with a soapy water solution. In the event of bubbles forming, the leak can be traced and sealed.
- Electronic pressure transducers and cables, plus connections to a computer data-logging facility via an analogue to digital converter. Static pressure transducers that can measure relative pressures of at least 120 kPa should be used. The pressure transducers should be calibrated by connecting them to a mercury manometer and comparing the electrical output to the actual pressure.
- Oxygen supply cylinder and regulator. It is recommended that the standard grade 99.8 % purity oxygen normally required for welding be used. The oxygen supply cylinder must be fitted with a regulator to regulate the flow of oxygen between the oxygen cylinder and the permeameters to the required 100 kPa starting pressure.
- Pressure gauges. Each permeameter must be fitted with a pressure gauge to serve as a check on the pressure recorded by the computer, or, in the case of manual readings, for taking actual measurements. Oxygen pressure gauges which do not contain oil or grease should be used, since these substances may react explosively with oxygen under pressure.

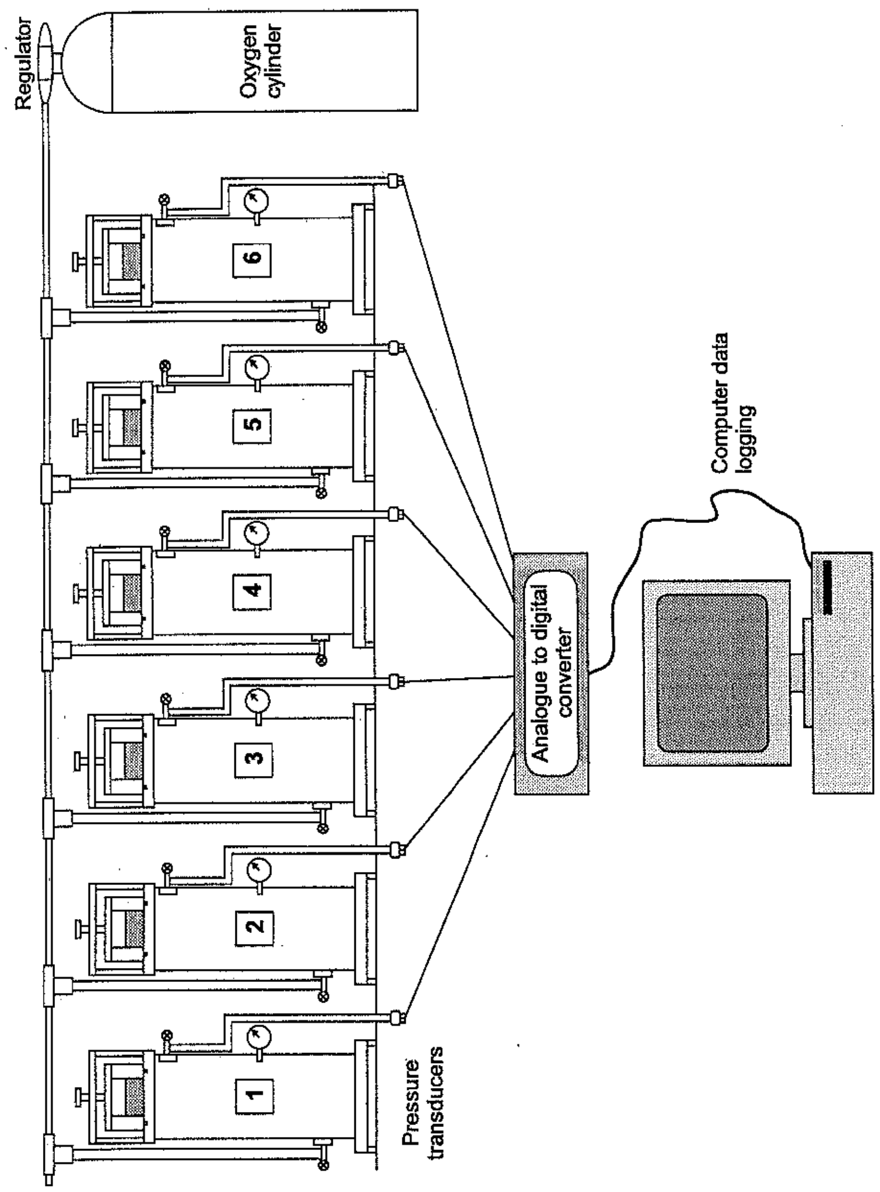

Figure 1.2: Oxygen permeability index test arrangement

- Silicone rubber collars. The concrete samples are placed in rubber collars that are fitted onto the permeability cells. The rubber collar is moulded as shown in Figure 1.3, using silicone rubber of Shore Hardness 39A. (Suitable materials are Silastic 9161 RTV silicone rubber with 2% 9162 catalyst, both manufactured by Dow Corning). The rubber mixture is poured into the mould, which should be lightly smeared with petroleum jelly, and left for 24 hours to set. With time the rubber collars may develop cracks due to the pressure applied when bolted into the permeability cell and it is recommended that the collars should be replaced from time to time to eliminate the chances of leakage. It is advised not to seal the collars in position with grease because of the danger of the oxygen under pressure reacting with the grease and causing an explosion.
- Valves and piping to connect the cells to the oxygen supply, and to allow isolation of each cell when under pressure.

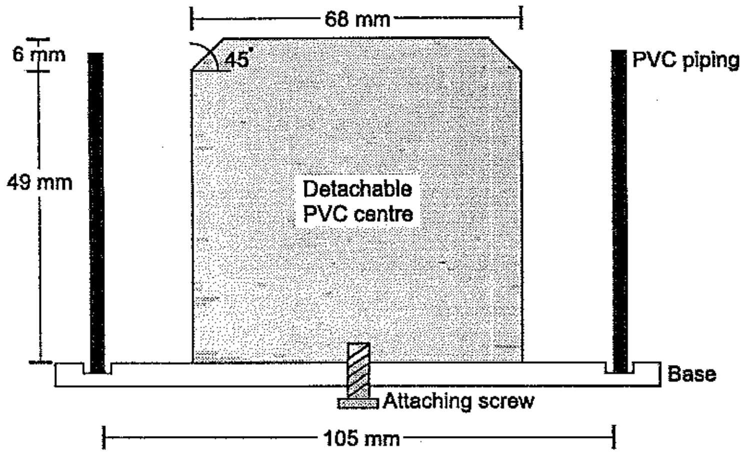

Figure 1.3: Cross section of the mould used for making silicone rubber collars

c) Vernier calliper to measure sample thickness.

d) Sample holding device, to permit safe and accurate cutting of the sample to length.

e) A water-cooled diamond tipped core barrel with an inner diameter of 68 mm, attached to a suitable coring drill.

f) A water-cooled moveable bed diamond saw.

g) Computer for data-logging, and analogue to digital converter.

Sample preparation

- Coring. The test samples used in the oxygen permeability test should be in the form of 25 ± 2 mm thick by 68 mm diameter concrete discs, cut from cores drilled from the original exposed face of a concrete member (or laboratory sample). (This thickness applies to concrete made with nominal 19 mm aggregate; for other coarse aggregate sizes, the thickness of the disc should be 5 mm greater than the maximum nominal aggregate size.) Place the core barrel at right angles to the concrete face and secure tightly in position. Core to a depth of approximately 60 to 80 mm. Carefully remove the core from the concrete face with a hammer and chisel. Mark each core and remove to the laboratory. (In the case of coring cubes, it is better to leave the core in the cube and cut the whole cube at the correct depth so as to separate the core.)
- Cutting. Secure the core tightly in the sample holding device on the moveable bed of the cutting machine. Slice off the outermost 5 mm of each core and discard. This is done to ensure that surface variations due to deposits from shutter release agents, surface unevenness etc. are excluded. Slice off the next 25 ± 2 mm section of the core and use this slice in the test. The top and bottom surfaces of the sample should be even, of equivalent cross-sectional area and as close to parallel as possible. If the same samples are to be used in the water sorptivity test after the oxygen permeability test, mark the faces of the samples corresponding to the external face of the core.

Both the coring and cutting operations must be done carefully and slowly to prevent damage to the samples.

Sample conditioning

a) Place the samples in an oven at 50 ± 2°C and at a relative humidity of less than 20 % for a minimum of 7 days.

b) After the drying period, remove the samples to be tested from the oven and leave to cool to room temperature for 1 hour on a dry steel surface in the room in which the testing is to be conducted. It is important that the samples are all tested immediately after the cooling period has expired in order to ensure that all the samples are at the same temperature and moisture content at the start of the test.

Test procedure

a) The permeability cell(s) should be housed in a room where the temperature is controlled at 23 ± 2°C and relative humidity at 60 ± 5%.

b) At the end of the cooling period, using a vernier calliper, measure the thickness of the sample at 4 equally spaced positions on the perimeter. These measurements should be recorded to the nearest 0.1 mm.

c) Place the sample in the silicone rubber collar and position this centrally on the top plate of the permeameter.

d) By tightening an appropriate screw arrangement, compress the rubber collar axially against the perforated plate. The amount of compression, monitored by the movement of the PVC spacer, should be between 6 mm and 10 mm. The spacer should be compressed equally on all sides to ensure that the sample does not skew in the collar allowing air to pass between the sample and the collar.

e) Open the inlet (lower) and outlet valves and allow oxygen from a regulated pressure cylinder to flow through the permeameter for a few seconds, in order to purge the permeameter.

f) Shut the outlet valve and allow the pressure to increase to approximately 100 kPa. Shut the inlet valve and start the computer data-logging (or alternatively manually record the first pressure reading). The computer should be set so that the time and pressure are recorded at predetermined time intervals.

g) Check the airtightness of the cell with soapy water solution.

h) Pressure readings should be recorded on the computer (or manually) at regular time intervals for 2 to 8 hours or longer, depending on the quality of the samples. For computer recording, the computer can be set beforehand to take readings at any time interval required. A reading every 5 to 10 minutes for the full period (defined as the period for the pressure to decay to about 50 kPa) ought to give enough data points through which to draw a straight line so that the regression coefficient is not less than 0.99. For manual recording, readings should be taken at 5 to 10 kPa decrements in pressure, down to a final pressure reading of about 50 kPa.

Calculation

The calculation involves conducting a linear regression analysis on the best-fit line obtained by plotting values of $\ln(P_0/P_t)$ against t, where P_0 is the initial pressure reading at the start of the test, and P_t are subsequent pressure readings at times t measured from the time of reading of initial pressure (time measured in seconds).

The regression analysis should give a correlation coefficient greater than 0.99. Where the correlation coefficient is less than 0.99, a re-test shall be done on the same sample. If it fails the re-test, the sample shall be discarded.

The coefficient of permeability is given by:

$$k = \frac{\omega V g d z}{R A \theta} \quad \text{.......} \quad (1.1)$$

where: k = coefficient of permeability of a test sample (m/s)
ω = molecular mass of oxygen (O_2) = 32 kg/mol
V = the volume of oxygen under pressure in the permeameter (m^3)
g = acceleration due to gravity (m/s^2)
R = universal gas constant = 8.313 (Nm/K mol)
A = superficial cross-sectional area of the sample (m^2)
d = sample thickness (m)
θ = absolute temperature (K)
z = slope of the line determined from the regression analysis above

The coefficient of permeability is calculated for each of the test samples.

The 'permeability index' is given by the negative log of the average of the coefficients of permeability of the samples, which, for four samples, is:

$$\text{Permeability index} = -\log_{10}[\tfrac{1}{4}(k_1 + k_2 + k_3 + k_4)] \quad \text{........} \quad (1.2)$$

Note that in general the higher the permeability index, the more resistant the concrete is to gas penetration.

Report

Report the following, if known:

c) Source of the specimen (or core)
d) Identification number of specimen (and core)
e) Location of specimen within core or cylinder
f) Type of concrete, including binder type, water/cement ratio, and other relevant data supplied with the samples
g) Description of specimen
h) Curing history of specimen
i) Unusual specimen preparation, for example, removal of surface treatment
j) Test results.

Precision and bias

Work still in progress.

Single operator precision

The single operator coefficient of variation of a single test result (three test determinations) has been found to be 2.5% (three specimens from same batch).

Multi-laboratory precision

Not done yet.

2. METHOD STATEMENT FOR THE WATER SORPTIVITY TEST FOR CONCRETE

Scope

The water sorptivity test is a uni-directional absorption test and involves placing a concrete sample, of which the sides are sealed, in a shallow layer of water. The test uses the mass of water absorbed from the bottom face of the sample as a measure of the sorptivity of the sample. The values will be stated in mm/√h (millimetres per square root of hour)

Summary of test method

This test method consists of measuring the mass of water absorbed with time from the bottom of a concrete sample, of which the sides have been sealed in order to ensure that water can only be absorbed from the bottom. The sorptivity is determined from the slope of the straight line produced when the mass of water absorbed is plotted against the square root of time, multiplied by the sample thickness, and divided by the difference between the vacuum saturated mass of the sample and the dry mass of the sample. One test result is calculated from the mean of three, or preferably four test determinations on the same material. It is recommended that at least one extra sample is prepared for the purpose of re-testing, in the case of a test failing.

Significance and use

a) This test method is suitable for evaluation of materials and mix proportions for design purposes as well as for research and development purposes.
b) This test method is suitable for use as a quality control test on construction projects, provided suitable statistical criteria are used to make judgements.
c) The details of the test method apply to 25 mm thick × 68 mm diameter specimens (normally taken from the cover layer of concrete). Other specimen thicknesses from 5 mm (mortar) to 40 mm may be used, but different conditioning times may be required.
d) Sample age may have significant effects on the test results, depending on the type of concrete and the curing procedure. Most

concretes, if properly cured, become progressively and significantly less absorptive with time.

e) Care should be taken in interpreting the result of this test when it is used on surface-treated concretes, or on concrete surfaces that have been exposed to environmental influences, such as carbonation or exposure to marine salts.

Apparatus

a) Plastic tray at least 20 mm deep and large enough to hold six of the samples being tested.

b) Absorbent paper towel.

c) Tap water saturated with calcium hydroxide.

d) Stopwatch.

e) Ventilated oven: controlled at 50 ± 2°C and a relative humidity below 20 %.

f) Weighing scale: accurate to 0.01 g.

g) Concrete coring (optional) and cutting equipment (of the water-lubricated diamond-tipped cutting type), including hammer and chisel.

h) Sample holding device, to permit safe and accurate cutting of the sample to length.

i) Vacuum saturation facility. A vacuum pump and chamber as shown in Figure 2.1 is needed for vacuum saturation of the samples. The vacuum pump must be able to create a vacuum of 75 ± 3 kPa. Glass flasks may be used as vacuum chambers. The glass tops of the flasks can be sealed onto the flasks with petroleum jelly applied to the contact area. Reservoirs with saturated $Ca(OH)_2$ solution must be fitted with screw-on tops to prevent the solution from evaporating. When opening the valve of the reservoir (while the chambers are under vacuum and sealed off from the rest of the system) the solution is automatically sucked into the chambers due to the vacuum. A pressure vessel that also serves as a catchment chamber preventing fluids from reaching the vacuum pump, must also be fitted into the system. The pressure vessel serves as a reservoir of vacuum in order to reduce the use of the pump. A pressure gauge that can read negative pressure must be fitted to the system.

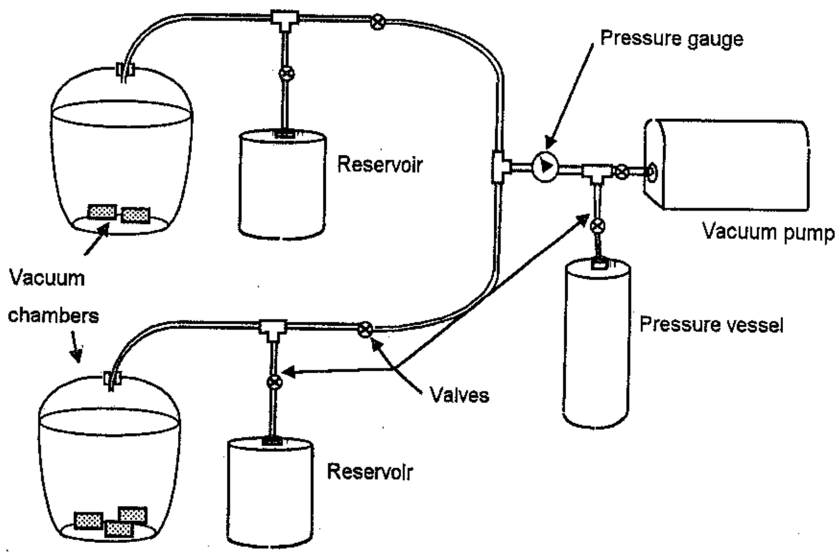

Figure 2.1: Suggested arrangement for the vacuum saturation facility

j) Epoxy paint and paintbrush. Use a paint that will produce a defect-free and waterproof coating on the sample. (Suitable products are ABE Epidermix 365 or Sikaguard Wetseal A + B.)

k) Vernier calliper, to measure sample thickness.

Test specimens

a) The water sorptivity test is designed to be conducted on up to 6 samples at any time. It is recommended that no more than 6 samples be tested at the same time, since the timing of the measurements is crucial in obtaining good results.

b) The same test specimens that were used in the oxygen permeability test may also be used in the water sorptivity test. After completion of the oxygen permeability test, the samples must be removed from the rubber collars and the vertical sides coated with epoxy paint as shown in Figure 2.2. This is done in order to ensure that the water will only be absorbed from the bottom face of the sample. Another way of achieving the same effect is to seal the cylindrical sides of the samples with tough plastic packaging tape. After sealing the sides of the samples, place the samples in the oven. After 24 hours in the oven, remove the samples to be tested and leave to cool on a dry steel surface for 1 hour in the room in which the testing is to be conducted.

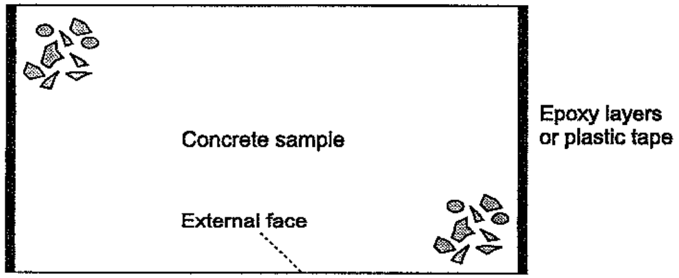

Figure 2.2: Epoxy coating on the side of the sample

c) In the case where the samples to be tested were not previously used in the oxygen permeability test, prepare samples as follows:
   - Coring. The test samples used in the water sorptivity test should be in the form of 25 ± 2 mm thick by 68 mm diameter concrete discs, cut from cores drilled from the original exposed face of a concrete member (or laboratory sample). Place the core barrel at right angles to the concrete face and secure tightly in position. Core to a depth of approximately 60 to 80 mm. Carefully remove the core from the concrete face with a hammer and chisel. Mark each core and remove to the laboratory. (In the case of coring cubes, it is better to leave the core in the cube and cut the whole cube at the correct depth so as to separate the core.)
   - Cutting. Secure the core tightly in the sample holding device on the moveable bed of the cutting machine. Slice off the outermost 5 mm of each core and discard. This is done to ensure that surface variations due to deposits from shutter release agents, surface unevenness etc. are excluded. Slice off the next 25 ± 2 mm section of the core and use this slice in the test. The top and bottom surfaces of the sample should be even, of equivalent cross-sectional area and as close to parallel as possible. Mark the face of the samples corresponding to the external face of the core. Both the coring and cutting operations must be done carefully and slowly to prevent damage to the samples.
   - Sample conditioning. Place the samples in an oven at 50 ± 2°C and at a relative humidity less than 20 % for a minimum of 7 days. After the drying period, remove the samples to be tested from the oven and leave to cool to room temperature. Coat the cylindrical sides with water-resistant epoxy paint as shown in Figure 2.2, and as described above. (Another way of achieving the same effect is to seal the cylindrical sides of the samples with tough plastic packaging tape.) After sealing the sides of the samples, place the samples in the oven. After 24 hours in the oven, remove the samples to be tested and leave to cool on a dry steel surface for 1 hour in the room in which the testing is to be conducted.

d) It is important that the samples are all tested immediately after the cooling period has expired in order to ensure that all the samples are at the same temperature and moisture content at the start of the test. It is also extremely important not to test samples until they have cooled to ambient temperature.

Test procedure

a) The water sorptivity test must be conducted in a room in which the temperature is controlled at 23 ± 1°C and the relative humidity at 60 ± 5 %.

b) During the cooling period, using a vernier calliper, measure the thickness of the samples at 4 equally spaced positions on the perimeter. These measurements should be recorded to the nearest 0.1 mm.

c) Place approximately 10 layers of absorbent paper in the plastic tray, leaving a gap of not less than 5 mm between the sides of the tray and the edges of the paper pad.

d) Pour sufficient $Ca(OH)_2$ saturated water into the tray such that a free surface of water is just visible above the surface of the paper pad. Smooth the paper pad out towards the edges in order to expel any trapped air bubbles.

e) When the cooling period has expired, weigh the samples and record this as the dry mass of the samples at time = 0, to an accuracy of 0.01 g.

f) Start the stopwatch and place the samples into the tray, one every 10 s, such that their "external" faces are on the wet paper pad as shown in Figure 2.3.

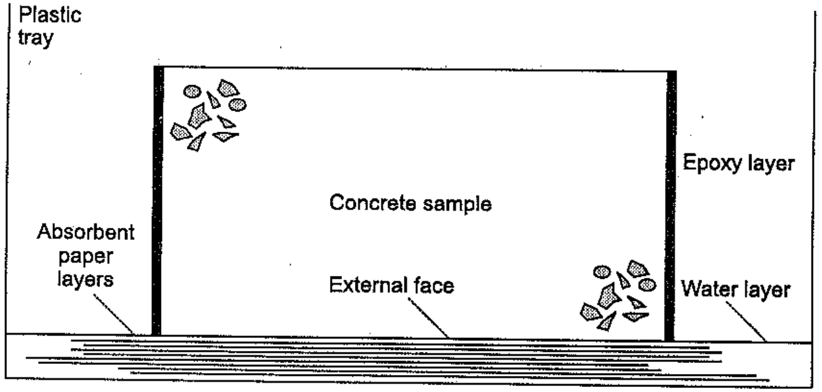

Figure 2.3: Water sorptivity test arrangement

g) As soon as the first minute after the first sample was placed in the tray has expired, the first sample must be removed from the tray, wiped once with a damp piece of absorbent paper, placed on the scale and the weight recorded accurately to the nearest 0.01 g. The first sample must then be placed back in the tray with its external face on the wet paper pad and the second sample taken from the tray, wiped once and placed on the scale. The process is then repeated until all samples are weighed and placed back in the tray. The process of weighing for each sample should take about 10 s. The weighing procedure is then repeated at intervals of 2, 4, 8, 16, 32, and, if necessary, 64 minutes after the first sample was originally placed onto the wet paper pad.

Note: 1) It is suggested that the number of measurements be increased to 8 or more for very dense concretes with low rates of absorption, in order to be able to draw proper regression lines through the points. It might also be appropriate to plot the values while testing, and then decide if it is necessary to increase the number of measurements taken.

2) The method given above for the process of weighing the samples has been found to be practical and simple to perform. A more rigorous method would involve stopping the stopwatch during the weighing operation for each sample. It is unlikely that there will be any practical difference between the results using the two methods. Further work is ongoing in this area.

h) The damp piece of absorbent paper with which the samples are wiped should be damp enough not to draw moisture from the samples when wiping, but not so wet as to leave the face of the sample with a water sheen still visible. If necessary the sheet is to be replaced with a new one during testing.

i) At the end of the weighing period, place the samples in the vacuum chamber and evacuate the chamber to a vacuum pressure of 75 ± 3 kPa. Maintain this vacuum for 2 hours. Isolate the chamber from the vacuum pump and, without opening the chamber, allow $Ca(OH)_2$ saturated water to flow into the chamber from the reservoir. When the water level is approximately 40 mm above the top of the samples, turn off the water flow and re-connect the chamber to the vacuum pump. Maintain the vacuum pressure at 75 ± 3 kPa for a further 24 hours.

j) At the end of the vacuum saturation period, remove the samples from the vacuum chamber, dry the surface using a paper towel and immediately weigh to an accuracy of 0.01g. Record this as the saturated weight of the sample.

Calculation

a) The mass of water absorbed at each weighing period (M_{wt}) is given by:

$$M_{wt} = M_{st} - M_{s0} \qquad (2.1)$$

where: M_{st} = the mass of the sample at time t
M_{s0} = the mass of the sample at time t = 0

b) By conducting a linear regression analysis, determine the value of F given by:

$$M_{wt} = F \times \sqrt{t} \quad \text{.......................} \quad (2.2)$$

where: F is the slope of the best-fit straight line obtained by plotting values of M_{wt} against $\sqrt{t}$, where t refers to the measurement times (in hours) after a sample is first exposed to water on its lower face.

From the regression analysis, record the value of F and the coefficient of correlation (R^2).

If the R^2 value is less than 0.99, it is recommended to plot the curve of M_{wt} against $\sqrt{t}$, and examine the curve for non-linear sections. These can arise due to inaccuracies at the start of a test, or due to saturation conditions being approached near the end of a test. Carry out further regression analyses as appropriate, selecting the most linear portion of the plot. However, at least 5 points are usually required to obtain a reliable regression analysis.

c) The water sorptivity (S) of the sample is given by:

$$S = \frac{F\ d}{M_{sv} - M_{s0}} \quad \text{.......................} \quad (2.3)$$

where: F = the slope of the regression line as determined above ($g/\sqrt{h}$);
d = the average of the 4 thickness measurements on the sample (mm);
M_{sv} = the vacuum saturated mass of the sample (g);
M_{s0} = the mass of the sample at time $t = 0$ (g).

Note that in general the lower the sorptivity value, the more resistant the concrete is to water penetration.

Report

Report the following, if known:

a) Source of the specimen (or core)
b) Identification number of specimen (and core)
c) Location of specimen within core or cylinder

d) Type of concrete, including binder type, water/cement ratio, and other relevant data supplied with the samples
e) Description of specimen
f) Curing history of specimen
g) Unusual specimen preparation, for example, removal of surface treatment
h) Test results.

Precision and bias

Work still in progress.

Single operator precision

The single operator coefficient of variation of a single test result (three test determinations) has been found to be 12% (three specimens from the same batch).

Multi-laboratory precision

Not done yet.

3. METHOD STATEMENT FOR THE CHLORIDE CONDUCTIVITY TEST FOR CONCRETE

Scope

The chloride conductivity test involves determining the DC conductivity of a concrete sample saturated with a sodium chloride solution. The test provides a rapid indication of the resistance of concrete to the penetration of chloride ions. The values will be stated in mS/cm (milliSiemens per centimetre).

Referenced documents

a) Streicher PE, and Alexander MG (1995). A chloride conduction test for concrete. Cement and Concrete Research, vol. 25, no. 6, pp. 1284-1294.

b) ASTM C1202-94. Standard test method for electrical indication of concrete's ability to resist chloride ion penetration. Amer. Soc. for Testing and Materials, 1994.

Summary of test method

The test method consists of measuring the electrical current passed through 25 mm thick slices of 68 mm diameter cores. The specimens are saturated with a 5 M NaCl solution before the test. A potential difference of 2, 5 or 10 V DC is applied cross the ends of the specimen, which is immersed in a sodium chloride solution. The conductivity is determined from the specimen dimensions and the specimen electrical resistance ((thickness/area) × (current/potential difference)). One test result is calculated from the mean of three, or preferably four test determinations on the same material. The conductivity is linearly related to the chloride diffusivity of the concrete by the diffusibility relationship (Streicher and Alexander (1995)).

Significance and use

a) This test is suitable for evaluation of materials and mix proportions for design purposes as well as research and development purposes.

b) This test is suitable for use as a quality control test on construction projects, provided suitable statistical criteria are used to make judgements.

c) The details of the test method apply to 25 mm thick × 68 mm diameter specimens. Specimens from 5 mm (mortar) to 40 mm thick may also be used, but different conditioning times may be required.

d) Sample age may have significant effects on the test results, depending on the type of concrete and the curing procedure. Most concretes, if properly cured, become progressively and significantly less permeable to chloride ions with time.

Interferences

Since the test results are a function of the electrical resistance of the specimen, the presence of reinforcing steel or other embedded electrically conductive materials may have a significant effect.

Apparatus

a) Oven capable of maintaining a temperature of 50 ± 2°C and a relative humidity below 20 %.

b) Vacuum saturation apparatus as shown in Figure 3.1 using the following:

- Vacuum pump. The pump must be capable of maintaining a vacuum of 75 ± 3 kPa.
- Pressure vessel/catchment chamber. The chamber must be approximately 25 *l* in volume to help maintain the vacuum when the pump is not running, with a pressure gauge (accurate to 0.5 kPa) that can read negative pressure connected between the pump and the cylinder. The vessel also acts as a water trap since the vacuum will be drawn over water. Should the vessel not be used the pump oil should be changed after each operation.
- Vacuum chambers. These should be approximately 4 *l* in volume and capable of withstanding vacuum pressures without imploding. The size of the cylinder will depend on the number of samples tested per run.

The vacuum saturation facility may also be used for preparing the samples for the water sorptivity test. The facility may be designed so that one set of a vacuum chamber and reservoir will contain the samples and sodium chloride solution for the chloride conductivity test, and the other set the samples and calcium hydroxide-saturated water for the sorptivity test. The reservoirs with prepared solutions must be fitted with screw-on tops to prevent the solutions from evaporating. When opening the valves of the reservoir (while the chambers are under vacuum and sealed off from the rest of the system) the solution is sucked into the chambers due to the vacuum. A pressure vessel that also serves as a catchment chamber preventing fluids from reaching the vacuum pump, must also be fitted into the system. The pressure vessel serves as a reservoir of vacuum in order to minimize the use of the pump.

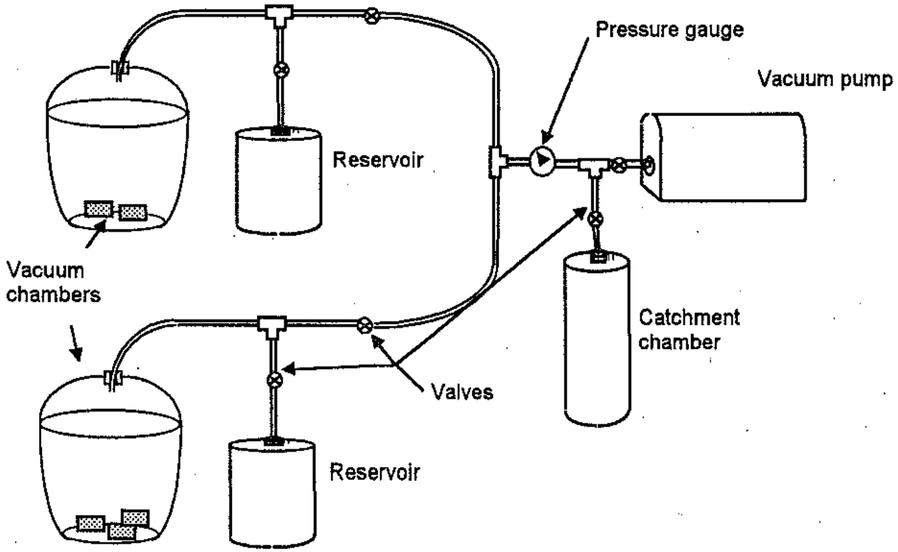

Figure 3.1: Vacuum saturation facility

c) A water-cooled diamond tipped core barrel with an inner diameter of 68 mm, attached to a suitable coring drill.

d) A water-cooled moveable bed diamond saw.

e) Vernier calliper to measure the specimen thickness.

f) Sample holding device, to permit safe and accurate cutting of the sample to length.

g) Conduction cell. The design is shown in Figure 3.2.

h) DC power source. 0 - 12 Volt, 0 - 1 Ampere stabilised.

i) Voltmeter and ammeter (2 multimeters). These must be digital 4 digit, 0 - 20 V range, 0 - 300 mA, rated accuracy 0.1 %.

j) Electrical cable and plugs.

k) Volumetric flask or container, a scale etc. to prepare the standard solutions, given below.

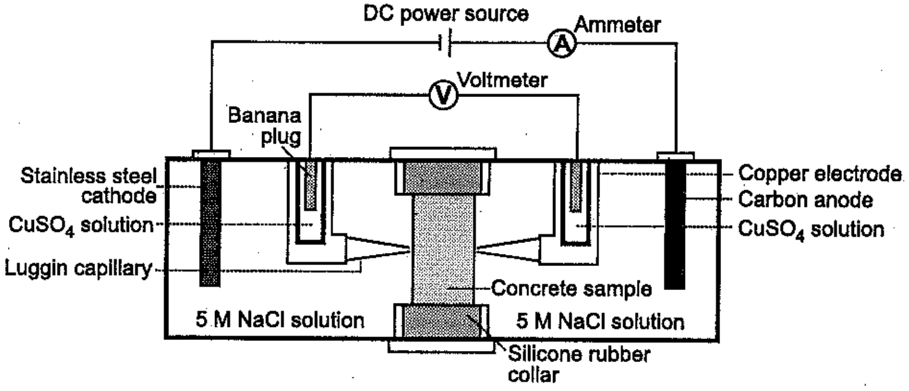

Figure 3.2: Conduction cell

Solutions, materials

a) Sodium chloride solution. This must be 5 M (± 0.1) NaCl CP grade in tap water (chlorides < 0.05%) that can be prepared as follows: A container marked at 10 *l* will be needed as well as 2.93 kg of CP grade NaCl (99 % purity). Add the salt to the container, add tap water but make sure not to exceed the 10 *l* level. Stir occasionally over a long period (1 day) until all the salt has dissolved. Top the container up to 10 *l* using tap water only after all the salt has dissolved. The container should be sealed to prevent evaporation. The solution can be heated to increase the rate of dissolution.

b) Saturated calcium hydroxide solution.

c) Saturated copper sulphate solution.

Sample preparation

The coring and cutting operations must be done carefully and slowly to prevent damage to the samples.

- Coring. The test samples used in the chloride conductivity test should be in the form of 25 ± 2 mm thick by 68 mm diameter concrete discs, cut from cores drilled from the original exposed face of a concrete member (or laboratory sample). Place the core barrel at right angles to the concrete face and secure tightly in position. Core to a depth of approximately 60 to 80 mm. Carefully remove the core from the concrete face with a hammer and chisel. Mark each core and remove to the laboratory. (In the case of coring cubes, it is better to leave the core in the cube and cut the whole cube at the correct depth so as to separate the core.)
- Cutting. Secure the core tightly in the sample holding device and on the moveable bed of the cutting machine. Slice off the outermost 5 mm of each core and discard. This is done to ensure that surface variations due to deposits from shutter release agents, surface unevenness etc. are excluded. Slice off the next 25 ± 2 mm section of the core and use this slice in the test. The top and bottom surfaces of the sample should be even, of equivalent cross-sectional area and as close to parallel as possible.

Sample conditioning

a) Place the samples in an oven at 50 ± 2 °C and at a relative humidity less than 20 % for a minimum of 7 days. After the drying period, remove the samples to be tested from the oven and leave to cool to room temperature for 1 hour on a dry steel surface in the room in which the testing is to be conducted.

b) Vacuum saturation. After the cooling period and before the chloride conductivity test, the samples are placed in the vacuum chamber, the chamber sealed and a vacuum of 75 ± 3 kPa drawn over the samples. The vacuum is maintained at 75 ± 3 kPa for three hours. This is done in order to remove air from the concrete pores, thereby facilitating the flow of solution into the concrete pores.

c) Immediately after the three hours have expired, the 5 M NaCl solution is allowed to enter the vacuum chamber and the samples and solution kept under a vacuum of 75 ± 3 kPa for five hours.

d) After the five hours have expired, the samples are left to soak in the NaCl solution for a further 18 ± 1 hrs.

Procedure

a) The chloride conductivity test must be done in a controlled temperature room at 23 ± 2°C.

b) It is best to execute the chloride conductivity test on a tray or in a container to minimise the risk of damage to the electronic equipment by spilt concentrated sodium chloride solution. Also, the operator should wear plastic gloves, since the strong sodium chloride solution is corrosive.

c) Fill both the cells with the 5 M salt solution, by first filling the Luggin capillaries and then the rest of the cell in order to prevent air-locks from forming in the capillaries.

d) The specimen is taken from the solution and placed in the centre of the silicone rubber collar inside the central section of the apparatus.

e) The cells containing the NaCl salt solutions are screwed onto the central section containing the specimen, thereby clamping the specimen by the lateral expansion of the silicone rubber collar. However, care must be taken not to deform the collar so much that it squashes over the sample, reducing the area through which the chloride ions will flow. On the other hand it must be tight enough so that ions do not pass between the sides of the sample and the collar. Petroleum jelly can be applied to the threads of the conduction cell in order to screw it on more easily.

f) Ensure that the copper halfcell electrodes are clean and that the halfcell tubes are filled with the saturated copper sulphate solution so as to ensure contact with the banana plugs.

g) The circuit is arranged as shown (Figure 3.2) and the current is measured at the applied voltage (2, 5 or 10 V). 10 V is preferred, but 2 or 5 V is used for very permeable or thin samples in order not to exceed the reading limits of the ammeter.

h) It is advisable to clean the conduction cell and equipment immediately after use in order to prevent the copper electrodes from rusting and salt crystals from clogging up the cell.

During the execution of the test an oxidation layer forms on the copper electrodes and they need to be cleaned periodically. A small piece of sandpaper can be used for this purpose, or the electrodes can be dipped into an acidic solution such as nitric acid. The banana plugs may also start rusting and can be cleaned with sandpaper. It is also advisable to clean all the equipment thoroughly with warm soapy water after a series of conductivity tests in order to minimise oxidation and clogging.

Calculation

To determine the conductivity of each sample, the measured current, applied voltage and the sample dimensions are substituted in equation 3.1:

$$\sigma = \frac{it}{VA} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (3.1)$$

where σ = conductivity of sample (mS/cm)
i = electrical current (mA)
V = potential difference (V)
t = thickness of sample (cm)
A = cross sectional area of sample (cm^2)

Three, or preferably four test determinations are averaged to obtain one test result.

Note that in general the lower the conductivity value, the more resistant the concrete is to chloride transport.

Report

Report the following, if known:

a) Source of the specimen (or core)
b) Identification number of specimen (and core)
c) Location of specimen within core or cylinder
d) Type of concrete, including binder type, water/cement ratio, and other relevant data supplied with the samples
e) Description of specimen
f) Curing history of specimen
g) Unusual specimen preparation, for example, removal of surface treatment

h) Test results.

Precision and bias

Work still in progress.

Single operator precision

The single operator coefficient of variation of a single test result (three test determinations) has been found to be 6 % for three specimens from the same batch, and 8 % for specimens from different batches.

Multi-laboratory precision

Not done yet.