



Standard Test Method for Determining the Apparent Chloride Diffusion Coefficient of Cementitious Mixtures by Bulk Diffusion¹

This standard is issued under the fixed designation C 1556; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers the laboratory determination of the apparent chloride diffusion coefficient for hardened cementitious mixtures.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

C 31/C 31M Practice for Making and Curing Concrete Test Specimens in the Field

C 42/C 42M Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete

C 125 Terminology Relating to Concrete and Concrete Aggregates

C 192/C 192M Practice for Making and Curing Concrete in the Laboratory

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

C 1152/C 1152M Test Method for Acid-Soluble Chloride in Mortar and Concrete

C 1202 Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration

2.2 NORDTEST Standards:

NT BUILD 443, Approved 1995-11, Concrete, Hardened: Accelerated Chloride Penetration (in English)³

¹ This test method is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.66 on Concrete's Resistance to Fluid Penetration.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Published by NORDTEST, P.O. Box 116 FIN-02151 ESPOO Finland, Project 1154-94, e-mail: nordtest@vtt.fi, website: <http://www.vtt.fi/nordtest>

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology C 125.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *apparent chloride diffusion coefficient, D_a , n* —a chloride transport parameter calculated from acid-soluble chloride profile data obtained from saturated specimens exposed to chloride solutions, without correction for chloride binding, that provides an indication of the ease of chloride penetration into cementitious mixtures.

3.2.2 *chloride binding, v* —the chemical process by which chloride ion is removed from solution and incorporated into cementitious binder hydration products.

3.2.2.1 *Discussion*—Chloride binding is primarily associated with hydration products formed by the aluminate phase of cement and mixtures containing ground granulated blast furnace slag.

3.2.3 *chloride penetration, v* —the ingress of chloride ions due to exposure to external sources.

3.2.4 *exposure liquid, n* —the sodium chloride solution in which test specimens are stored prior to obtaining a chloride profile.

3.2.5 *exposure time, n* —the time that the test specimen is stored in the solution containing chloride ion.

3.2.6 *initial chloride-ion content, C_i , n* —the ratio of the mass of chloride ion to the mass of concrete for a test specimen that has not been exposed to external chloride sources.

3.2.7 *profile grinding, v* —the process of grinding off and collecting a powder sample in thin successive layers from a test specimen using a dry process.

3.2.8 *surface chloride content, C_s , n* —the theoretical ratio of the mass of chloride ion to the mass of concrete at the interface between the exposure liquid and the test specimen.

4. Summary of Test Method

4.1 Obtain a representative sample of the cementitious mixture prior to exposure to chloride ion. Separate each sample into a test specimen and an initial chloride-ion content specimen. Crush the initial chloride-ion content specimen and determine the initial acid-soluble chloride-ion content. Seal all sides of the test specimen, except the finished surface, with a

*A Summary of Changes section appears at the end of this standard.

suitable barrier coating. Saturate the sealed specimen in a calcium hydroxide solution, rinse with tap water, and then place in a sodium chloride solution. After a specified exposure time, the test specimen is removed from the sodium chloride solution and thin layers are ground off parallel to the exposed face of the specimen. The acid-soluble chloride content of each layer is determined. The apparent chloride diffusion coefficient and the projected surface chloride-ion concentration are then calculated using the initial chloride-ion content, and at least six related values for chloride-ion content and depth below the exposed surface.

5. Significance and Use

5.1 This test method is applicable to cementitious mixtures that have not been exposed to external chloride ions, other than the negligible quantity of chloride ion exposure from sample preparation using potable water, prior to the test.

5.2 The calculation procedure described in this test method is applicable only to laboratory test specimens exposed to a sodium chloride solution as described in this test method. This calculation procedure is not applicable to specimens exposed to chloride ions during cyclic wetting and drying.

NOTE 1—The diffusion of ionic species in concrete occurs within the fluid-filled pores, cracks and void spaces. The concentration and valence of other ionic species in the pore fluid also influence the rate of chloride diffusion, and therefore, the apparent diffusion coefficient as determined by this test procedure.

5.3 In most cases, the value of the apparent chloride diffusion coefficient for cementitious mixtures changes over time (See Note 2). Therefore, apparent diffusion coefficients obtained at early ages may not be representative of performance in service.

NOTE 2—The rate of change of the apparent diffusion coefficient for cementitious mixtures containing pozzolans or blast-furnace slag is typically different than that for mixtures containing only portland cement.

5.4 The apparent chloride diffusion coefficient is used in Fick's second law of diffusion to estimate chloride penetration into cementitious mixtures that are in a saturated condition.

5.5 The apparent chloride diffusion coefficient is commonly used in chloride ingress models based on Fick's second law of diffusion. The apparent diffusion coefficient determined by this method includes chemically bound chloride, so proper use of the apparent chloride diffusion coefficient to predict chloride ingress requires consideration of chloride binding.

5.6 The resistance to chloride penetration is affected by such factors as the environment, finishing, mixture composition, workmanship, curing, and age.

6. Apparatus

6.1 *Balance*, accurate to at least ± 0.01 g.

6.2 *Thermometer*, accurate to at least ± 1.0 °C.

6.3 *Controlled Temperature Laboratory or Chamber*. The laboratory or chamber shall maintain the temperature of a water bath at 23 ± 2 °C.

6.4 *Plastic Container*, with tight-fitting lid. Select a container size in accordance with provisions in 9.1.2.

6.5 Equipment for grinding off and collecting powder from concrete, mortar, or grout specimens in layers of approximately 2 mm thickness. Refer to Figs. 1 and 2 for examples of satisfactory equipment (See Note 3).

NOTE 3—A lathe or milling machine equipped with a short-barrel carbide-tipped, or diamond-tipped, core drill bit has been found satisfactory for profile grinding.

6.6 *Resealable Polyethylene Bags*, 200- to 300-mm wide by 250- to 300-mm long, and sheet thickness not less than 0.1 mm.

6.7 Equipment for crushing concrete, mortar or grout. Suitable equipment is described in Test Method C 1152/C 1152M.

6.8 Equipment for chloride analysis as described in Test Method C 1152/C 1152M.

6.9 *Slide Caliper*, accurate to at least ± 0.1 mm.

7. Reagents and Materials

7.1 *Distilled or De-ionized Water*.

7.2 *Calcium Hydroxide* [$\text{Ca}(\text{OH})_2$], technical grade.

7.3 *Calcium Hydroxide Solution*, saturated, (approx. 3 g/L).

7.4 *Sodium Chloride* [NaCl], technical grade.

7.5 *Exposure Liquid*—An aqueous NaCl solution prepared with a concentration of 165 ± 1 g NaCl per L of solution.

7.6 *Two-component Polyurethane or Epoxy-resin Based Paint*, capable of forming a barrier membrane that is resistant to chloride ion diffusion.

8. Test Specimens

8.1 Drilled cores, molded cylinders, or molded cubes are acceptable test specimens. One sample consists of at least two test specimens representative of the cementitious mixture under test (See Note 4). Specimens must be free of defects such as voids or cracks visible to the unaided eye (See Note 5). The minimum dimension across the finished surface of each test specimen must be at least 75 mm, but not less than three times the nominal maximum aggregate particle size. The specimen depth must be at least 75 mm.

NOTE 4—The material between the exposed surface and the outermost layer of reinforcement is often of interest because it is here that the protection against chloride penetration is needed. Furthermore, the quality of the material in this particular area can deviate from that in the rest of the system, as this region is often affected by construction practices.

NOTE 5—Specimens with voids deeper than the profile layer thickness can increase the apparent rate of chloride penetration, and increases test variability.

8.2 Unless otherwise specified, provide 28 days of laboratory standard moist curing in accordance with Practice C 31/ C 31M or C 192/C 192M prior to sample preparation for immersion in the exposure liquid.

8.2.1 Describe any variance from standard curing practice in the report.

8.3 For drilled cores obtained according to Test Method C 42/C 42M, prepare the test specimen by cutting off the outermost 75 mm of the core. The test specimen thus obtained has one face that is the original finished surface, and the other face that is a sawn surface as shown in Fig. 3.

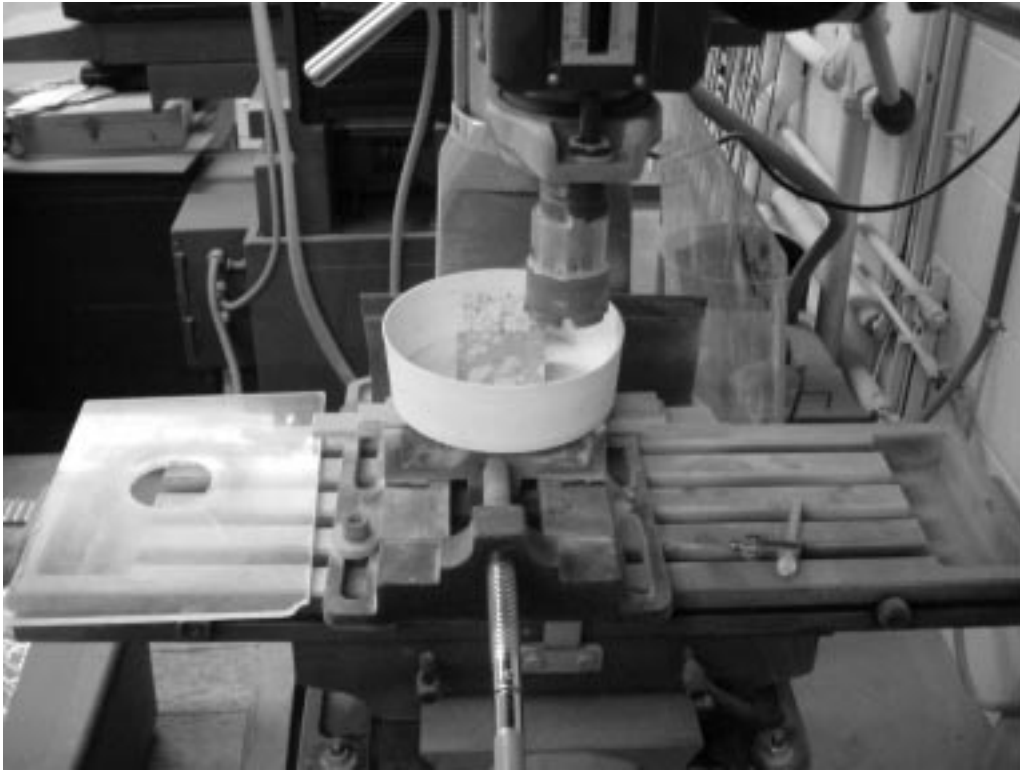


FIG. 1 Profile Grinding Using a Milling Machine

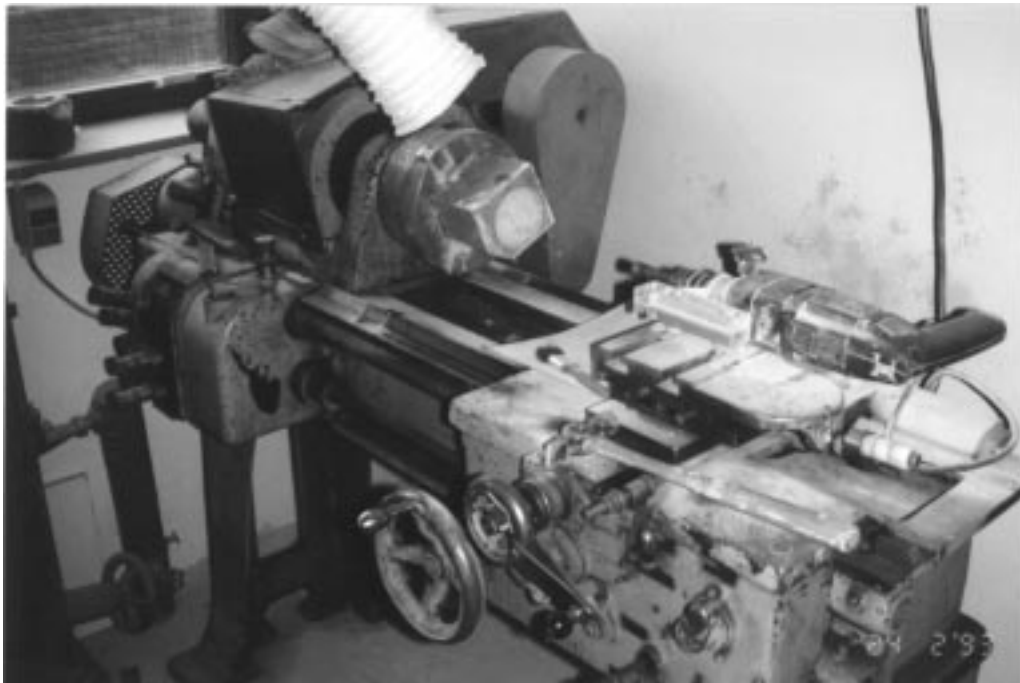


FIG. 2 Profile Grinding Using a Lathe

8.4 For specimens prepared in accordance with Practice C 31/C 31M or C 192/C 192M, the test specimen is prepared by cutting parallel to the finished surface. The top 75 mm is used as the test specimen (see Fig. 3).

8.5 From the remainder of the drilled core, or molded specimen, cut a slice that is at least 20-mm thick. Use this slice to determine the initial chloride-ion content, C_i .

8.6 Rinse the specimens with tap water immediately after cutting. Scrub the surface with a stiff nylon brush, and rinse again. Prior to sealing specimen surfaces, air dry until no moisture can be removed from the surface with a dry paper towel (See Note 6).

8.6.1 Exposure specimens must be surface-dry but internally moist prior to sealing. This condition is satisfied by

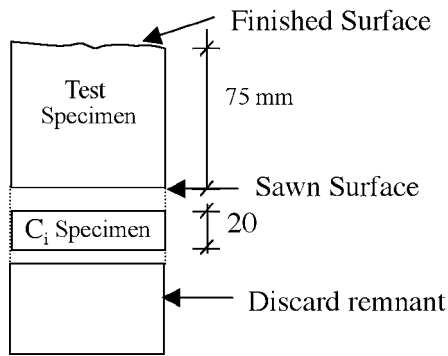


FIG. 3 Sketch of Specimens Obtained from a Typical Sample

standard moist-cured specimens allowed to air dry for no more than 24 h in laboratory air maintained at 23 ± 2 °C and 50 ± 3 % RH.

NOTE 6—Specimens cured in a saturated calcium hydroxide water bath are normally covered by residual lime particles. If this residue is not removed and test specimens are allowed to temporarily dry in air, a calcium carbonate layer can form on the surface of the specimen. This carbonate layer may interfere with the test result, which is why cleansing and rinsing with tap water after cutting or removal from the saturated calcium hydroxide water bath is required.

8.7 Seal all sides of the exposure specimen except for the finished surface following the procedure described in Test Method C 1202.

8.8 Determine the initial mass of the test specimen when the coating has hardened.

8.9 Immerse the test specimen in the saturated calcium hydroxide water bath at 23 ± 2 °C in a tightly closed plastic container. The container must be filled to the top to prevent carbonation. After 24 h of immersion, remove the specimen, blot the surface dry with a paper towel, and determine the mass of the specimen in the surface-dry condition.

8.10 The test specimen is immersed in a saturated calcium hydroxide water bath until the mass does not change by more than 0.1 % in 24 h (See Note 7). An acceptable alternative procedure is to vacuum saturate the specimens with saturated calcium hydroxide solution using a vacuum chamber similar to the system described in Test Method C 1202.

NOTE 7—Typically, the mass of moist-cured specimens stabilizes within 48 h.

9. Procedure

9.1 Exposure:

9.1.1 Remove the saturated test specimen from the calcium hydroxide water bath, immediately rinse the specimen surface with tap water, place the specimen in the exposure container, fill the container with the exposure liquid, and then seal the container. Place the container in a temperature-controlled chamber or room maintained at 23 ± 2 °C. Record the start date and start time to the nearest hour.

9.1.2 It is permitted to place multiple specimens in a single container as long as the specimens are placed in the container such that the entire exposure surface is unobstructed. Maintain the exposed surface area to exposure liquid volume ratio within the range of 50 ± 30 cm²/L (See Note 8).

NOTE 8—The volume of exposure liquid required for nominal 100-mm diameter cylinder or core exposure specimens is approximately one liter per specimen.

9.1.3 The specimens must remain in the exposure liquid for at least 35 days (See Note 9).

NOTE 9—The exposure time should be extended for mixtures such as those that are more mature, were made with low w/cm, or high-performance mixtures containing supplementary cementitious materials.

9.1.4 If evaporation of water from the exposure liquid or a container leak allows the specimen surface to dry during the exposure time, the test is not valid (See Note 10).

NOTE 10—It is suggested to monitor the mass of the sealed container if evaporation of water from the exposure solution is expected.

9.1.5 Record the exposure time to the nearest hour.

9.2 Profile Grinding:

9.2.1 Remove the test specimen from the exposure liquid, rinse with tap water, and dry for at least 24 h in laboratory air maintained at 23 ± 2 °C and 50 ± 3 % RH.

9.2.2 When grinding is to be performed longer than 48 h after removal from the exposure solution, store the specimens in watertight resealable polyethylene bags until time of grinding. When grinding is to be performed longer than 7 days after removal from the exposure solution, store the bagged specimens in a freezer maintained at -15 °C (± 5 °C) until time of grinding.

9.2.3 Obtain the powder samples by grinding off material in layers parallel to the exposed surface. Do not grind closer than

TABLE 1 Recommended Depth Intervals (in mm) for Powder Grinding^A

w/cm	0.25	0.30	0.35	0.40	0.50	0.60	0.70
Depth 1	0-1	0-1	0-1	0-1	0-1	0-1	0-1
Depth 2	1-2	1-2	1-2	1-3	1-3	1-3	1-5
Depth 3	2-3	2-3	2-3	3-5	3-5	3-6	5-10
Depth 4	3-4	3-4	3-5	5-7	5-8	6-10	10-15
Depth 5	4-5	4-6	5-7	7-10	8-12	10-15	15-20
Depth 6	5-6	6-8	7-9	10-13	12-16	15-20	20-25
Depth 7	6-8	8-10	9-12	13-16	16-20	20-25	25-30
Depth 8	8-10	10-12	12-16	16-20	20-25	25-30	30-35

^A Luping, Tang and Sørensen, Henrik, "Evaluation of the Rapid Test Methods for Measuring the Chloride Diffusion Coefficients of Concrete," NORDTEST Project No. 1388-98, Swedish National Testing and Research Institute, SP Report 1998:42.

NOTE—For cementitious mixtures with pozzolan or slag, the depth intervals in the column one place to the left should be applied. For example, use the depth intervals for w/cm = 0.35 for silica fume concrete with w/cm = 0.40.

5 mm from the edge of the specimen to avoid edge effects and disturbances from the coating.

9.2.4 For the minimum exposure time of 35 days, grind off eight layers in accordance with Table 1. For longer exposure times, select depth increments such that a minimum of 6 points span the range from 1 mm below the exposed surface to a depth with a chloride-ion content equal to, or slightly greater than, the initial chloride-ion content.

9.2.5 The following alternate profiling procedure is permitted if the exposure time is sufficient to allow chloride penetration deeper than 40 mm. Slice the test specimen parallel to the exposure surface using a water-cooled diamond saw in 5- to 6-mm increments, minimizing the time specimens are exposed to water. Dry the slices for 24 h in laboratory air, then crush and prepare the powder sample as described in Test Method C 1152/C 1152M.

9.2.6 Obtain a sample of at least 10 g of powder from each layer. Determine the distance from the exposure surface to the mid-depth of each layer. For example, the layer thickness and mid-depth are determined from measurements of the specimen before and after powder sample collection. Calculate the depth below the exposed surface as the average of five uniformly distributed measurements using a slide caliper.

9.3 Chloride Analysis:

9.3.1 Determine the acid-soluble chloride-ion content of the powder samples, C_x (mass %), to ± 0.001 % according to Test Method C 1152/C 1152M.

9.3.2 Obtain the initial chloride-ion content, C_i (mass %), from the 20-mm thick slice by crushing and prepare a powder sample as described in Test Method C 1152/C 1152M.

9.4 Record any deviations from the requirements of this method.

10. Calculations

10.1 Test Results:

10.1.1 Determine the values of surface concentration and apparent chloride diffusion coefficient by fitting Eq 1 to the measured chloride-ion contents by means of a non-linear regression analysis using the method of least squares. Omit the chloride-ion content determined from the exposure surface layer in the regression analysis. All other chloride-ion content measurements are included in the regression analysis.

$$C(x,t) = C_s - (C_s - C_i) \cdot \operatorname{erf}\left(\frac{x}{\sqrt{4 \cdot D_a \cdot t}}\right) \quad (1)$$

where:

$C(x,t)$ = chloride concentration, measured at depth x and exposure time t , mass %,

C_s = projected chloride concentration at the interface between the exposure liquid and test specimen that is determined by the regression analysis, mass %,

C_i = initial chloride-ion concentration of the cementitious mixture prior to submersion in the exposure solution, mass %,

x = depth below the exposed surface (to the middle of a layer), m,

D_a = apparent chloride diffusion coefficient, m^2/s ,
 t = the exposure time, s, and

erf = the error function described in Eq 2.

$$\operatorname{erf}(z) = 2 / \sqrt{\pi} \cdot \int_0^z \exp(-u^2) du \quad (2)$$

10.1.2 Tables with values of the error function are given in standard mathematical reference books.⁴ The error function is also included as a library function in most electronic calculation software.

10.1.3 The test results are:

10.1.3.1 The initial chloride concentration, C_i (mass %), stated to three significant digits.

10.1.3.2 The projected surface chloride concentration at the exposed surface, C_s (mass %), stated to three significant digits.

10.1.3.3 The apparent chloride diffusion coefficient, D_a (m^2/s), stated to 2 significant digits.

10.2 *Non-linear Regression Analysis*—Perform the regression analysis by minimizing the sum given in Eq 3. Refer to Fig. 4 for clarification.

$$S = \sum_{n=2}^N \Delta C^2(n) = \sum_{n=2}^N (C_m(n) - C_c(n))^2 \quad (3)$$

where:

S = sum of squares to be minimized, (mass %)²,

N = the number of layers ground off,

$\Delta C(n)$ = difference between the measured and calculated chloride concentration of the n th layer, mass %,

$C_m(n)$ = measured chloride concentration of the n th layer, mass %, and

$C_c(n)$ = calculated chloride concentration in the middle of the n th layer, mass %.

10.3 Other Calculations:

10.3.1 Plot the measured chloride contents at all points versus depth below the surface. Plot the best-fit curve on the same graph (see Fig. 4).

11. Report

11.1 Report the following information if known:

11.1.1 Name and address of the laboratory, and the place at which tests were performed, if different from the laboratory address.

11.1.2 Date and identification number of the test report.

11.1.3 Method of sampling and other circumstances (date and person responsible for sampling).

11.1.4 Description of the tested object including specimen type, identification marks, mixture proportions, the date the tested object was cast, curing regimen employed, and age at the start of exposure.

11.1.5 Start date and duration of the exposure time.

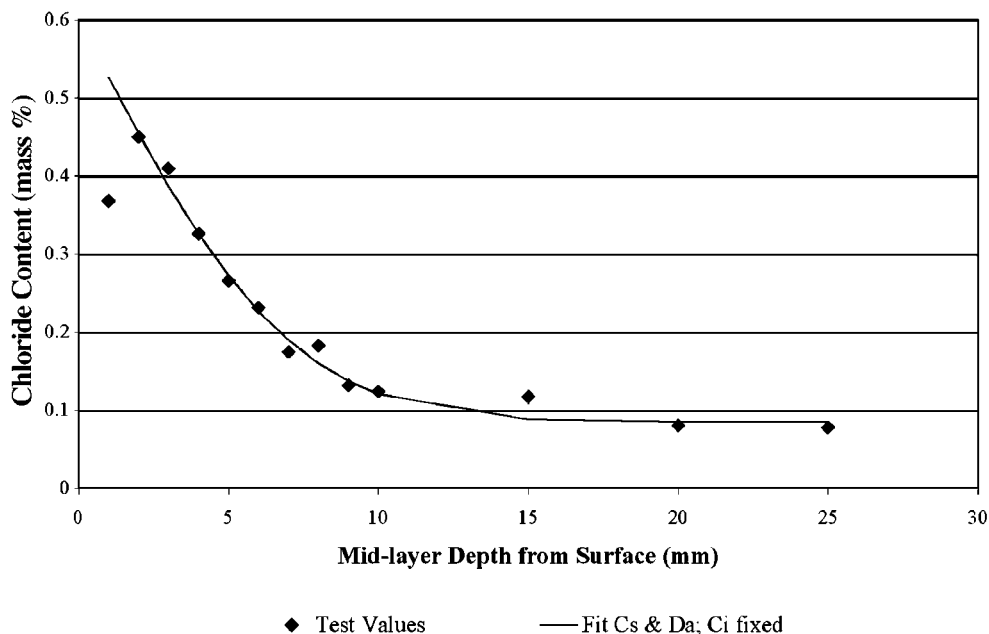
11.1.6 Conditioning of the test specimens, and a description of the exposure conditions during the test, such as temperature, evidence of evaporation.

11.1.7 Identification of the test equipment and instruments used.

11.1.8 Any deviation from the test method together with other information of importance for judging the result.

⁴ Beyer, W. H., ed., *CRC Handbook of Mathematical Sciences*, 5th Edition, CRC Press, Boca Raton, FL, 1978.

Chloride Profile Fit Using
 $C_{x,t} = C_s - (C_s - C_i) * (\text{erf}(x/\sqrt{4Dt}))$



◆ Test Values — Fit C_s & D_a ; C_i fixed
FIG. 4 Sample Regression Analysis

TABLE 2 Example Calculation

C_s (mass %)	C_i (mass %)	D_a (m^2/s)	t (yr)	Sum (Error) ²
0.605	0.085	4.86E-13	1.00	2.2151E-03

x (mm)	Measured Value	Predicted Value	Error, $\Delta C(n)$ (Meas.-Pred.)	(Error) ²
1	0.368	0.530		
2	0.450	0.458	-8.19E-03	6.72E-05
3	0.410	0.391	1.94E-02	3.76E-04
4	0.326	0.329	-3.31E-03	1.10E-05
5	0.266	0.275	-9.49E-03	9.01E-05
6	0.231	0.230	1.25E-03	1.55E-06
7	0.175	0.192	-1.71E-02	2.93E-04
8	0.183	0.162	2.08E-02	4.34E-04
9	0.132	0.139	-7.07E-03	5.00E-05
10	0.124	0.122	2.16E-03	4.66E-06
15	0.117	0.089	2.85E-02	8.12E-04
20	0.080	0.085	-5.16E-03	2.66E-05
25	0.078	0.085	-7.00E-03	4.90E-05

11.1.9 A table listing the chloride-ion content measurements of each layer and mid-depth for each layer.

11.1.10 A plot showing the measured chloride-ion contents for each layer and the best-fit curve from the regression analysis.

TABLE 3 Precision Estimates^A

Coefficient	Statistic	Single Laboratory	Multiple Laboratory
D_a	CV ^B	14.2	20.2
	d2s %	39.8	56.6
C_s	CV	13.3	18.1
	d2s %	37.2	50.7

^A These statistics represent the CV (1s %) and d2s % statistics as defined in Practice C 670.

^B Coefficient of variation.

11.1.11 The measured value of C_i and the values of C_s , and D_a determined from the regression analysis.

11.1.12 Date and signature.

12. Precision and Bias

12.1 *Precision*—There has been no interlaboratory study of this test method. However, there are precision data⁵ from an interlaboratory study of NORDTEST NT Build 443, from which this test method was developed. The report includes data from two interlaboratory studies involving three concrete mixtures and three to five laboratories participated, depending on the mixture. Average values of D_a among the mixtures ranged from 2.1 to $14.7 \times (10^{-12}) m^2/s$. Average values of C_s among the mixtures ranged from 0.61 to 1.0%. Table 3 summarizes the single-laboratory and multiple-laboratory coefficient of variation and maximum difference expected between duplicate determinations in 95 % of such comparisons. Therefore, the apparent diffusion coefficient results of two properly conducted tests should not differ by more than 39.8 % of the mean value.

12.2 *Bias*—Since there is no accepted reference material suitable for determining the bias of this test method, no statement on bias is made.

13. Keywords

13.1 chloride; concrete; corrosion; diffusion; ion transport; service life

⁵ Luping, Tang and Sørensen, Henrik, "Evaluation of the Rapid Test Methods for Measuring the Chloride Diffusion Coefficients of Concrete," NORDTEST Project No. 1388-98, Swedish National Testing and Research Institute, SP Report 1998:42.

SUMMARY OF CHANGES

Committee C09 has identified the location of selected changes to this test method since the last issue, C 1556 – 03, that may impact the use of this test method. (Approved June 1, 2004)

(1) Added Note 1 to Significance and Use, increment reference numbers for all existing notes. (2) Freezing requirement added to 9.2.2.

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