



Designation: **C1556 – 11a (Reapproved 2016) C1556 – 22**

## Standard Test Method for Determining the Apparent Chloride Diffusion Coefficient of Cementitious Mixtures by Bulk Diffusion<sup>1</sup>

This standard is issued under the fixed designation C1556; 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 ( $\epsilon$ ) 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 standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

C31/C31M Practice for Making and Curing Concrete Test Specimens in the Field

C42/C42M Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete

C125 Terminology Relating to Concrete and Concrete Aggregates

C192/C192M Practice for Making and Curing Concrete Test Specimens in the Laboratory

C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

C1152/C1152M Test Method for Acid-Soluble Chloride in Mortar and Concrete

C1202 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)<sup>3</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology C125.

<sup>1</sup> 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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<sup>2</sup> 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.

<sup>3</sup> 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

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

### 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 test surface, with a 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 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  $\pm 0.01$  g.

6.2 *Thermometer*, accurate to at least  $\pm 1.0$  °C.

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

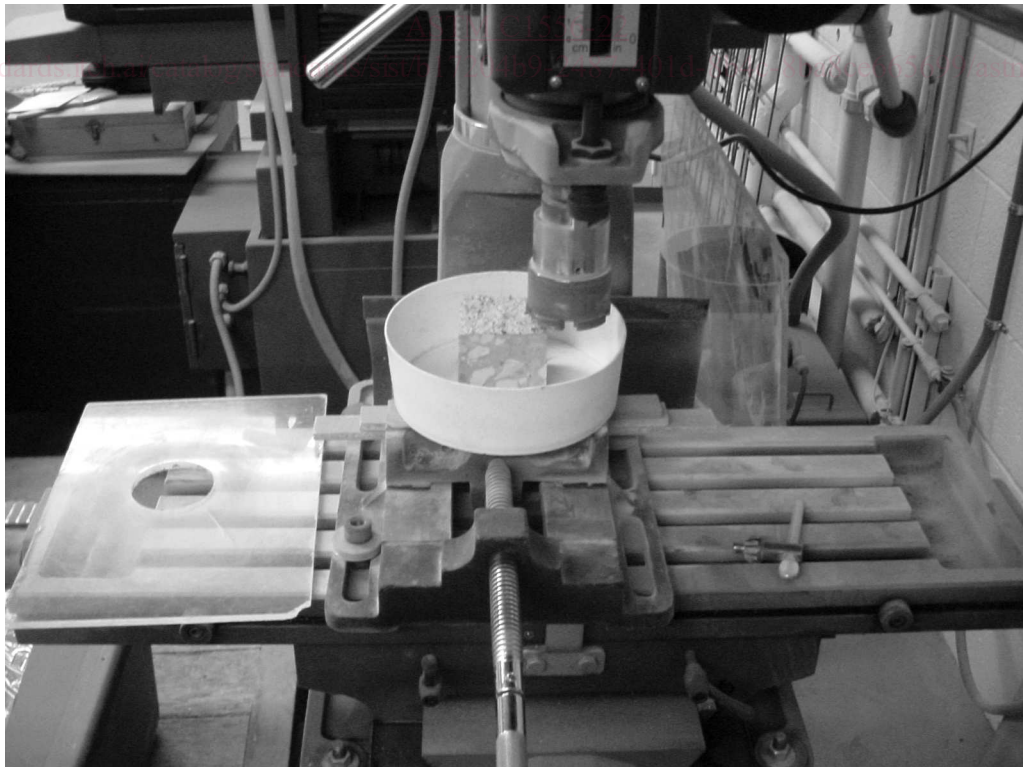


FIG. 1 Profile Grinding Using a Milling Machine

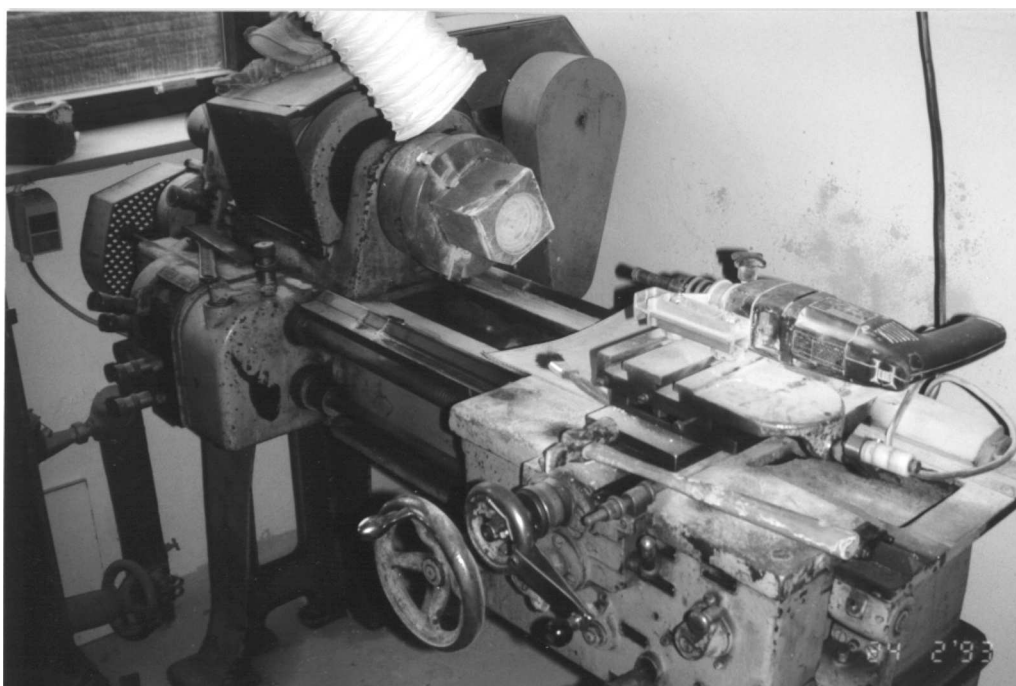


FIG. 2 Profile Grinding Using a Lathe

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6.6 *Resealable Polyethylene Bags*, ~~200-200 mm~~ 200 mm to ~~300-mm~~ 300 mm wide by ~~250-250 mm~~ 250 mm to ~~300-mm~~ 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 **C1152/C1152M**.

6.8 Equipment for chloride analysis as described in Test Method **C1152/C1152M**.

6.9 *Slide Caliper*, accurate to at least  $\pm 0.1$  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* [ $\text{NaCl}$ ], technical grade.

7.5 *Exposure Liquid*—An aqueous NaCl solution prepared with a concentration of ~~165~~ 165 g  $\pm$  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 test. **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 exposed 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—Specimens with voids deeper than the profile layer thickness can increase the apparent rate of chloride penetration, and increases test variability.

NOTE 5—The material quality of the concrete 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 C31/C31M or C192/C192M 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 from structures not previously exposed to chlorides according to Test Method C42/C42M, prepare the test specimen by cutting off and discarding the outermost 75 mm-50 mm of the core. core (see Note 5The 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-). The next segment of at least 75 mm thickness is used as the test specimen with the exposed surface at 50 mm depth from the top of the original cast specimen (see Fig. 3).

8.4 For specimens prepared in accordance with Practice C31/C31M or C192/C192M, the test specimen is prepared by cutting parallel to the finished surface. The top 75 mm-50 mm is cut off and discarded. The next segment of at least 75 mm thickness is used as the test specimen with the exposed surface at 50 mm depth from the top of the original cast 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-20 mm thick. Use this slice to determine the initial chloride-ion content,  $C_i$  either by crushing the entire slice or by grinding off a layer at least 2 mm-2 mm thick. Alternately, if the profile from the diffusion test specimen is ground deep enough such that the last 2 successive layers taken have chloride contents within 0.01 % by mass of concrete of each other, it is permitted to extrapolate the best-fit equation of the chloride profile to obtain the initial chloride-ion content,  $C_i$ .

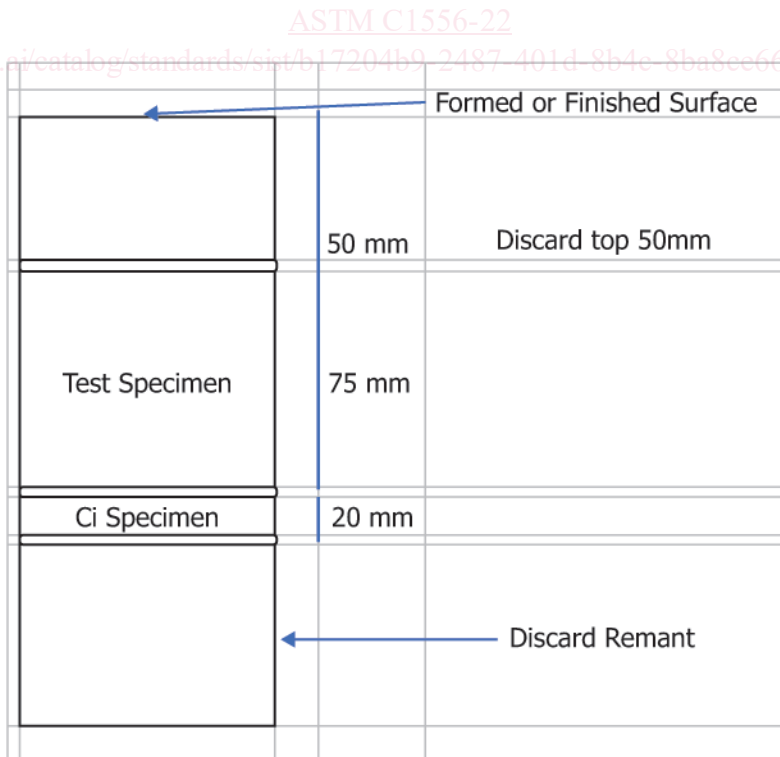


FIG. 3 Sketch of Specimens Obtained from a Typical Sample Not Previously Exposed to Chlorides



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 standard moist-cured specimens allowed to air dry for no more than 24 h in laboratory air maintained at  $23 \pm 2$  °C and  $50 \pm 3$  % 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~~exposed surface following the procedure described in Test Method **C1202**.

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  $23 \pm 2$  °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 **C1202**.

**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  $23 \pm 2$  °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  $50 \pm 30$  cm<sup>2</sup>/L (see **Note 8**).

**NOTE 8**—The volume of exposure liquid required for nominal ~~100-mm~~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  $23 \pm 2^\circ\text{C}$  and  $50\text{--}50\% \text{RH} \pm 3\% \text{RH}$ .

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  $-15^\circ\text{C} (\pm 5^\circ\text{C})$  until time of grinding.

9.2.3 Obtain the powder samples by grinding off material in layers parallel to the exposed surface. Unless the coating is removed by sawing or by using a chisel, do not grind closer than 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 at least 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 **C1152/C1152M**.

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  $\pm 0.001\%$  according to Test Method **C1152/C1152M**.

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 **C1152/C1152M**.

9.4 Record any deviations from the requirements of this method.

## 10. Calculations

### 10.1 Test Results:

**TABLE 1 Recommended Depth Intervals (in mm) for Powder Grinding<sup>A</sup>**

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

<sup>A</sup> 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 1—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  $w/cm = 0.40$ .