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Admixtures for concrete, mortar and grout - Test methods - Part 14: Determination of the effect on corrosion susceptibility of reinforcing steel by potentiostatic electro-chemical test

Zusatzmittel für Beton, Mörtel und Einpressmörtel - Prüfverfahren - Teil 14: Bestimmung des Korrosionsverhaltens von Stahl in Beton - Elektrochemische Prüfung bei gleichbleibendem Potential

Adjuvants pour béton, mortier et coulis - Méthodes d'essais - Partie 14 : Détermination de l'effet sur la tendance a la corrosion de l'acier pour armature au moyen d'un essai électrochimique potentiostatique

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English Version

Admixtures for concrete, mortar and grout - Test methods - Part
14: Determination of the effect on corrosion susceptibility of
reinforcing steel by potentiostatic electro-chemical test

Adjuvants pour béton, mortier et coulis - Méthodes d'essai -
Partie 14 : Détermination de l'effet sur la tendance à la
corrosion de l'acier pour armature au moyen d'un essai
électrochimique potentiostatique

Zusatzmittel für Beton, Mörtel und Einpressmörtel -
Prüfverfahren - Teil 14: Bestimmung des
Korrosionsverhaltens von Stahl in Beton -
Elektrochemische Prüfung bei gleich bleibendem Potential

This European Standard was approved by CEN on 19 August 2006.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 480-14:2006) has been prepared by Technical Committee CEN/TC 104 "Concrete and related products", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2007, and conflicting national standards shall be withdrawn at the latest by April 2007.

This document has been prepared by Subcommittee 3 (SC 3) of TC 104 "Admixtures of concrete."

This document is part of the series EN 480 "Admixtures for concrete, mortar and grout - Test methods" which include the following:

Part 1: Reference concrete and reference mortar for testing

Part 2: Determination of setting time

Part 4: Determination of bleeding of concrete

Part 5: Determination of capillary absorption

Part 6: Infrared analysis

Part 8: Determination of the conventional dry material content

Part 10: Determination of water soluble chloride content

Part 11: Determination of air void characteristics in hardened concrete

Part 12: Determination of the alkali content of admixtures

Part 13: Reference masonry mortar for testing mortar admixtures

Part 14: Determination of the effect on corrosion susceptibility of reinforcing steel by potentiostatic electrochemical test

This document is applicable together with the standards of the series EN 934 "Admixtures for concrete, mortar and grout.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This European Standard defines:

A test method for determining the influence of an admixture on the corrosion of a steel bar embedded in a mortar sample held at an increased potential in an electrochemical cell. The method can be used to determine the harmlessness of admixtures with regard to corrosion of reinforcing but not stressed steel. It applies to all admixtures for concrete, mortar and grout in contact with reinforcing steel.

NOTE 1 This test method does not apply to assessing corrosion behaviour of admixtures when used with high tensile steel.

NOTE 2 This test method does not assess the efficacy of admixtures defined as corrosion inhibitors.

NOTE 3 The quantity of chloride ion present in admixtures is covered by a separate requirement in the relevant parts of the EN 934 series.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 196-1, *Methods of testing cement – Part 1: Determination of strength*

EN 197-1, *Cement – Part 1: Composition, specifications and conformity criteria for common cements*

3 Test principle

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During the test, a steel bar, embedded in a cement mortar containing the admixture, is maintained at a constant potential relative to a reference electrode. The potential chosen is close to the higher level of the passive state for steel in plain concrete. Any detrimental effect from the addition of the admixture will be immediately obvious as the passive state will change, leading to increased anodic dissolution rates and corrosion currents above those of the control. The test specimen containing the admixture, is compared with a control specimen without admixture, to assess the effect that the admixture has on the corrosion process.

4 Equipment, materials and test specimens

4.1 Equipment and materials

4.1.1 Electrodes

4.1.1.1 Working electrode: comprising a smooth bar, without visible scratches or corrosion, of low carbon mild steel (carbon less than 0,4 %, steel tensile strength > 300 MPa), e.g. S235JRG1 to EN 10025-1 and 10025-2. The working electrode is cylindrical, (7 ± 1) mm in diameter and the length in contact with mortar is ≥ 65 mm (see Figure 1).

4.1.1.2 Reference electrode: saturated calomel electrode, silver/silver chloride electrode or mercury/mercuric oxide electrode.

4.1.1.3 Counter electrode: platinum electrode or a thin sheet of stainless steel (e.g. EN 10088-1 type X2CrNi19-11) with a submerged length equal to the length of the working electrode in contact with the mortar.

4.1.2 Reference mortar

The composition of the reference mortar shall conform to EN 196-1. The cement shall be a CEM I in accordance with EN 197-1 and C_3A content $\leq 3,0$ %; water shall be de-ionized and the water/cement ratio shall be 0,50. Preparation of the mortar shall be in accordance with EN 196-1. For each test, two mixes are prepared: a reference control mortar, without admixture, and a reference test mortar which contains the admixture or admixture constituent being tested at the maximum recommended dose. All mortar is made using materials from the same delivery. If a liquid admixture is being tested, it is added to the mixing water of the test mortar and the amount of mixing water is decreased by the water content of the admixture. If a powder/granulated admixture is being tested it is added to the sand for the test mortar.

NOTE In some cases it may be difficult to obtain a dense, uniform and fully compacted mortar specimen when using the admixture at its maximum recommended dose under the specified test conditions. Segregation, excessive retardation, air entrainment or poor compaction due to early stiffening all have to be avoided as they may affect the test result. In such cases, modifications to the mixing and test procedures such as test and/or curing temperature or time/method of filling the mould should be agreed with the manufacturer and recorded in the test report. In exceptional cases, when testing retarding admixtures it may be necessary to increase the mix and curing temperature.

When testing set accelerating admixtures for sprayed concrete use a $w/c = 0,55$, cool the constituents to $(5 \pm 1) ^\circ C$ and add the admixture 15 s before the end of mixing.

4.1.3 Moulds

Moulds used in the preparation of a particular set of specimens (control and test) shall be made from cardboard, plastic or steel and essentially identical in internal dimensions. A suitable arrangement, including the dimensional requirement, is shown in Figure 1.

4.1.4 Test cell solution

The electrolyte of the cell shall be a saturated calcium hydroxide solution at a temperature of $(20 \pm 2) ^\circ C$. The electrolyte shall be made from analytical grade calcium hydroxide in de-ionised water.

4.1.5 Polarisation circuit

The polarisation circuit includes:

- potentiostat to apply a potential between the working electrode and the reference electrode, maintained at $(+ 500 \pm 5)$ mV_{NHE}. The potentiostat provides a constant voltage between the working electrode and the

counter electrode relative to the reference electrode. The potentiostat should have a minimum impedance of 100 M Ω .

- ammeter, with an accuracy $\leq 0,1 \mu\text{A}$, to measure changes in the current between the working electrode and the counter electrode. The range of the ammeter should be between 0 μA and $x \mu\text{A}$ where $x = 100$ times exposed surface of the working electrode in cm^2 .

4.2 Preparation of test specimens

4.2.1 General

Condition all the materials at $(20 \pm 2) ^\circ\text{C}$ before preparing the specimens.

NOTE Following the note in 4.1.2 a different conditioning temperature may be necessary when some admixtures are tested at their maximum recommended dose.

4.2.2 Preparation of the working electrode

All steel bar working electrodes used in a particular set of tests shall be from the same heat. Before making the reinforced mortar specimen, clean the working electrode using metallographic emery paper, grade 400 and then degrease with acetone or other suitable solvent. Handle the bars (working electrodes) with gloved hands only from this point in the procedure. Immediately after cleaning, coat each working electrode with an appropriate epoxy resin for a length of approximately 40 mm centred about the point where it emerges from the mortar (see Figure 1). Measure the length and diameter of the uncoated part of the electrode to be encased in mortar to the nearest 1,0 mm and 0,1 mm respectively.

NOTE 1 If the working electrode is not incorporated in mortar immediately after cleaning and coating, store it in a desiccator at a relative humidity $\leq 30 \%$. If the surface has become tarnished it should be cleaned again before use.

NOTE 2 Other coatings can be used in place of epoxy resin if they can be shown to give equivalent results.

4.2.3 Preparation of the mortar specimens

Prepare three similar specimens from each mortar mix.

Place the working electrode centrally in the mould so that the mortar cover to the embedded part without coating is (20 ± 5) mm and secure it to prevent movement during filling, compaction and whilst the mortar remains in the plastic state. Fill the moulds and compact to ensure complete compaction without segregation. If the mortar is not fully compacted or is subject to segregation, this can induce corrosion and invalidate the test. In such cases the test shall be repeated, if necessary with a change to the test conditions as suggested in the note in 4.1.2.

NOTE The working electrode may be placed in the mould after filling, provided it can be centrally located and full contact between the mortar and the bar achieved.

After preparation store the specimens at $(20 \pm 2) ^\circ\text{C}$ and $\geq 95 \%$ RH for 24 h prior to demoulding. If the specimens do not harden in 24 h, demould after hardening and record the age for inclusion in the test report.

5 Testing

5.1 Preparation for testing

After demoulding place the specimen vertically into a test cell, fill with the test cell solution (clause 4.1.4), to within 10 mm of the top of the mortar cylinder, and leave for 24 h at $(20 \pm 2) ^\circ\text{C}$. Do not allow water or the test cell solution to come into contact with the top surface of the specimen at any time as moisture around the embedded bar may lead to errors in the test.

5.2 Test procedure

After the 24 h equilibration period, thoroughly clean the exposed end of the working electrode then connect the working electrode, reference electrode and the counter electrode to the polarisation circuit as shown in Figure 2.

Raise the output of the potentiostat, until the working electrode potential, referred to the normal hydrogen reference electrode, reaches $(+500 \pm 5) \text{ mV}_{\text{NHE}}$. Maintain this potential for at least 24 h whilst continuously recording the current between the working electrode and the counter electrode.

Keep the top of the test specimen dry as contact with water or the test cell solution can lead to errors in the test result.

After completing the 24 h test period, break open the test specimens and examine the working electrodes for crevice corrosion at the boundary of the protective coating. If evidence of crevice corrosion is found discard the specimens and repeat the test. If there is no crevice corrosion examine the surface of the working electrode which was in contact with the mortar. If there is evidence that corrosion has occurred as a result of voids caused by poor compaction or due to segregation, discard the specimens and repeat the test. Take photographic records of any corrosion.

5.3 Test result

For each test specimen, calculate the surface area of the working electrode which is in direct contact with the mortar including the end section.

Read off the maximum current between 1 h and 24 h, and calculate the maximum current density as $\mu\text{A}/\text{cm}^2$ using the calculated area of the rebar (working electrode) in contact with mortar.

6 Test report

The test report shall include:

- full details of the composition of the mortar used for reference control and reference test specimens;
- admixture or constituent and dosage;
- details of the curing process for each specimen;
- type of counter electrode;
- for each specimen:
 - exposed surface of the working electrode;
 - maximum current density as $\mu\text{A}/\text{cm}^2$ of working electrode surface occurring between 1 h and 24 h;
 - plot of current density versus time;
 - visual inspection including any photographs of corrosion and confirmation that there is no crevice or other corrosion arising from deficiencies, such as poor compaction or segregation in the test specimen.