SLOVENSKI STANDARD
SIST EN 480-10:1998

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Kemijski dodatki za beton, malto in injekcijsko maso - Metode preskuġanja - 10. del: Doloļ anje deleġa vodotopnih kloridov

Admixtures for concrete, mortar and grout - Test methods - Part 10: Determination of water soluble chloride content

Zusatzmittel für Beton, Mörtel und Einpreßmörtel - Prüfverfahren - Teil 10: Bestimmung des wasserlöslichen Chloridgehaltes

Adjuvants pour béton, mortier et coulis - Méthodes d'essai - Partie 10: Détermination de la teneur en chlorure soluble dans l'eau

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ICS:

91.100.10	Cement. Mavec. Apno. Malta	Cement. Gypsum. Lime. Mortar
91.100.30	Beton in betonski izdelki	Concrete and concrete products

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EUROPEAN STANDARD

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English version

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Zusatzmittel für Beton, Mörtel und Einpreßmörtel - Prüfverfahren - Teil 10: Bestimmung des wasserlöslichen Chloridgehaltes

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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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Contents

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 104 "Concrete (performance, production, placing and compliance criteria)", 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of an identical text or by endorsement, at the latest by March 1997, and conflicting national standards shall be withdrawn at the latest by March 1997.

This standard is applicable together with the other standards of the series EN 480 for testing admixtures according to the series EN 934.

This Standard series EN 480 consists of the following parts:

Part 1: Reference concrete and reference motar 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

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard describes methods for determining water soluble halogens (exept fluorides) in admixtures.

The total water soluble halogen content is expressed as the chloride content.

2 Principle

The object of the test is to determine the content of chloride ions (including other halogen ions except fluorides) in an admixture by precipitation of the chloride ions with a silver nitrate solution, according to the reaction:

$$Cl^- + Ag^+ \rightarrow AgCl \qquad \text{(solid)}$$

The end point of this reaction is determined with the aid of a pH meter.

The volume of the sample required for this test has been calculated for an admixture of which the chloride content is lower than 0,1 % by mass. If the chloride content is known, or can be assumed to be higher, the admixture has to be diluted in a precise ratio before carrying out the test.

3 Apparatus

3.1 pH meter

Either:

a) a pH millivoltmeter in which case the graph is plotted point by point, or

b) a recording pH millivoltmeter connected to an automatic burette.

3.2 Electrodes

Either:

a) a combination of two electrodes consisting of:

an indicator: silver

a reference: mercurous sulphate (electrolyte KNO_3) or calomel with algar gel (electrolyte KNO_3) or

b) a combined electrode (indicator and reference) Ag - $AgCl$ (electrolyte KNO_3).

NOTE: Other electrodes such as ion selective electrodes may be used provided the results will be unaffected.

3.3 Additional apparatus

a) Balance, with an accuracy of 0,1 mg reading up to 200 g

b) 20 ml burette graduated with an accuracy of 0,05 ml

c) 250 ml and 500 ml beakers

d) 1000 ml measuring flask

e) heating device with a magnetic stirrer

f) 10 ml, 20 ml and 50 ml graduated flasks.

3.4 Reagents

All reagents shall be of analytical grade.

- Dilute nitric acid; mix equal volumes of concentrated nitric acid (d = 1,38) and distilled water
- Hydrogen peroxide at 110 volumes
- 33 % sodium hydroxide solution
- Ethanol
- pre-prepared 0,01 mol/l silver nitrate solution normality n determined to 0,0001

4 Procedure

4.1 Preparation of sample

4.1.1 Liquid admixture

From a liquid admixture a sample of (10 ± 1) g is weighed to 0,01 g and placed into a 250 ml or a 500 ml beaker.

4.1.2 Powder admixture

From a powder admixture a sample of (5 ± 1) g is weighed to 0,01 g. The sample is placed into a 250 ml or a 500 ml beaker.

4.2 Determination

4.2.1 General

Depending upon the composition of the admixture one of the following methods shall be used.

4.2.2 Method 1

If the admixture does not contain constituents that will interfere with the test, such as lignosulphonates, thiocyanates or reducing agents, dilute the sample or dissolve it and make the solution up to 100 ml with distilled water. Then add 5 ml of dilute nitric acid and carry out the titration of the chloride ions with the silver nitrate solution, recording the volume (V) used to 0,05 ml.

Carry out a blank titration under the same conditions and record the volume (V_o) to 0,05 ml.

4.2.3 Method 2

If the admixture does contain, or can be assumed to contain, lignosulphonates or reducing agents, the sample shall be diluted or dissolved and made up to 100 ml with distilled water. Then add 5 ml of sodium hydroxide solution and 10 ml of hydrogen peroxide.

In order to avoid loss of chloride ions make sure that the pH value remains above 8,5 by adding, if necessary, more sodium hydroxide solution. Bring the solution very slowly to the boiling point by means of a heating device with a magnetic stirrer(*e*) and boil for 30 min [1]).

Leave this solution to cool to (20 ± 2) °C. Then add 10 ml of the dilute nitric acid. Check whether the pH value is below 5,0. If not add more of the dilute nitric acid.

Carry out the titration of the chloride ions using the silver nitrate solution, recording the volume (V) used to 0,05 ml.

Carry out a blank titration under the same conditions and record the volume (V_o) to 0,05 ml.

4.2.4 Method 3

NOTE: This test should be carried out under an extractor to minimize the risk of escape of hydrogen cyanide.

If the admixture does contain or can be assumed to contain thiocyanate, place the sample in a 500 ml beaker in order to avoid losses by overflow. Carefully add 10 ml of hydrogen peroxide. This will cause an extremely exothermic reaction. After this reaction has subsided add again 10 ml of hydrogen peroxide. This shall be repeated three times.

Heat the solution slowly to the boiling point and keep it boiling for 2 h. Leave the solution to cool slowly to (20 ± 2) °C.

Then transfer the solution into a 250 ml beaker with distilled water and proceed according to 4.2.3 commencing with the adding of 10 ml diluted nitric acid.

5 Results

The results shall be expressed in % by mass.

$$\text{Chloride content} = \frac{3{,}55\,(V - V_o)\,n}{m}\ \%\ \textit{by mass}$$

where

V volume of silver nitrate solution used in the test in millilitres

V_o volume of silver nitrate solution used in the blank titration in millilitres

n normality of silver nitrate solution in mol per litre

m mass of the sample in gramms.

6 Test report

For a liquid admixture, if the chloride ion content is less than 0,005 % state "chloride ion content less than 0,005 %".

For a powder admixture, if the chloride ion content is less than 0,01 %, state "chloride ion content less than

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[1]) Certain admixtures may foam whilst boiling. This may be reduced by the addition of ethanol.

In other cases the chloride ion content shall be reported to the nearest 0,01 %.

The method used for testing shall be indicated in the report, i. e. "tested according to method '1', '2' or '3' ".

If method 2 or 3 has been applied, the volume of hydrogen peroxide, the boiling time as well as any other comments deemed necessary, shall also be stated.

NOTE: All three methods give the total halogen ion content (except fluoride) as chloride. In the case of method 2, any thiocyanate will also be included as chloride.