SLOVENSKI STANDARD
SIST EN 480-12:1998

01-maj-1998

Kemijski dodatki za beton, malto in injekcijsko maso - Metode preskuġanja - 12. del: Doloļ anje deleġa alkalij v kemijskih dodatkih

Admixtures for concrete, mortar and grout - Test methods - Part 12: Determination of the alkali content of admixtures

Zusatzmittel für Beton, Mörtel und Einpreßmörtel - Prüfverfahren - Teil 12: Bestimmung des Alkaligehaltes von Zusatzstoffen

Adjuvants pour béton, mortier et coulis - Méthodes d'essai - Partie 12: Détermination de la teneur en alcalins dans les adjuvants

Ta slovenski standard je istoveten z: **EN 480-12:1997**

ICS:

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

SIST EN 480-12:1998 **en**

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EN 480-12

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ICS 91.100.10; 91.100.30

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English version

Admixtures for concrete, mortar and grout - Test methods - Part 12: Determination of the alkali content of admixtures

Adjuvants pour béton, mortier et coulis - Méthodes d'essai - Partie 12: Détermination de la teneur en alcalins dans les adjuvants

Zusatzmittel für Beton, Mörtel und Einpreßmörtel - Prüfverfahren - Teil 12: Bestimmung des Alkaligehalts von Zusatzstoffen

This European Standard was approved by CEN on 3 April 1997.

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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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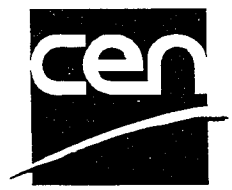

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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 Standard had been prepared by Subcommittee 3 (SC 3) of TC 104 "Admixtures for concrete, mortar and grout".

The present standard is applicable together with the standards of the series EN 480 and EN 934.

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 1998, and conflicting national standards shall be withdrawn at the latest by April 1998.

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

1 Scope

This European Draft Standard specifies a method for the determination of the alkali (sodium and potassium) content of admixtures for concrete, mortar and grouts in accordance with the series EN 934.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Draft Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 196-21
Methods of testing cement – Determination of the chloride, carbon dioxide and alkali content of cement

EN 934-2
Admixtures for concrete, mortar and grout – Part 2: Concrete admixtures – Definitions and requirements

ISO 648
Laboratory glassware – One-mark pipettes

ISO 1042
Laboratory glassware – One-mark volumetric flasks

3 Principle

An atomic absorption spectrophotometer is used to measure the sodium and potassium in dilute nitric acid extracts from admixtures. The extract is nebulised into an air/acetylene flame and the absorption of sodium or potassium radiation as it passes through the flame is measured. The amount of absorption is directly proportional to the sodium or potassium content of the extract at low concentrations. The sodium and potassium contents are separately measured and their sum, proportioned by molecular mass, is reported as the total equivalent Na_2O (alkali) content.

As an alternative to an atomic absorption spectro-photometer a suitable calibrated flame photometer can be used for the determination of sodium and potassium in the test solutions prepared in accordance with this standard.

4 Reagents

a) Concentrated nitric acid, analytical reagent grade 1 : 1.

b) Distilled or deionised water. The same source of water shall be used for preparation of calibration solutions and sample extracts.

c) Sodium stock solution (100 mg Na_2O per litre). Dissolve 0,188 g sodium chloride, analytical reagent grade, dried at (105 ± 5) °C to constant mass, in water (4b), dilute to 1 litre in a volumetric flask (5b)) with water[1)].

d) Potassium stock solution (100 mg K_2O per litre). Dissolve 0,158 g potassium chloride, analytical reagent grade, dried at (105 ± 5) °C to constant mass, in water (4b), dilute to 1 litre in a volumetric flask (5b)) with water[1)].

e) Calibration solution[2)]. Measure the required volume of stock solution shown in table 1 with a pipette and nitric acid with a plastic measuring cylinder. Transfer to a 1 litre graduated volumetric flask and dilute to the mark with water. Mix thoroughly and transfer to a clean dry plastic bottle with a water and air tight closure as described in 5e.

Table 1: Calibration solutions

Na_2O and K_2O concentration mg/l	Sodium stock solution (4c) ml	Potassium stock solution (4d) ml	Nitric Acid (4a) ml
0	0	0	10
2,5	25	25	10
5	50	50	10

5 Apparatus

a) Balance with a capacity of up to 200 g readable to 0,1 mg.

b) 100 ml and 1 litre volumetric flasks of class A in accordance with ISO 1042.

c) Calibrated pipettes of class A in accordance with ISO 648.

d) 10 ml and 25 ml measuring cylinders.

e) 1 litre plastic bottles with water and air tight closure. The plastic shall be such that no sodium or potassium is leached into 1 % nitric acid solution over a period of 6 months.

f) Atomic absorption spectrophotometer (AAS) equipped with burners suitable for air/acetylene flames and with a combination Na/K hollow cathode lamp or separate Na and K lamps.

g) A flame photometer (according to EN 196-21) if alternatively used in accordance with clause 3.

6 Preparation of calibration graphs

Operate the AAS or the flame phometer in accordance with the manufacturer's instructions. Whilst spraying water adjust the wave length setting around 589 nm (for Na) to give the maximum signal on the detector.

Spray the 5,0 mg/l calibration solution and adjust burner height and acetylene flow to give maximum absorption. Spray the 0 mg/l solution and adjust the gain control to give 0 % absorption and then in turn spray the

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[1)] Commercially available calibrated solutions for spectroscopic applications may be used.

[2)] The volumes of stock solutions given in table 1 may not be appropriate when commercially abailable spectroscopic solutions are used. In addition some atomic absorption spectrophotometers may require a smaller rang of Na_2O and K_2O concentration for calibration and use. In this case modify the calibration solutions accordingly.

other calibration solutions and record the absorption for each one.

Repeat the operation at a wavelength setting around 768 nm (for K).

Some instruments may be used in direct readout concentration mode. When this procedure is used follow the manufacturer's calibration instructions.

7 Test procedure

7.1 Preparation of test solution

7.1.1 Liquid admixture

Mix the samples thoroughly and transfer approximately 1 ml to a pre-weighed weighing boat. Record the mass of admixture sample in mg to the nearest 0,1 mg (m_1).

Transfer to a 1 litre volumetric flask (5b) with 150 to 250 ml water (4b) and add 10 ml nitric acid. Dilute to the mark with water. Mix thoroughly.

7.1.2 Powder admixtures

Mix the sample thoroughly and weigh approximately 1 g recording the actual mass in mg to 0,1 mg (m_1). Transfer to a 500 ml glass beaker, add 150 to 250 ml water (4b)) and 10 ml nitric acid (4a)). Heat to near boiling on a hot plate and maintain at near boiling for (15 ± 5) minutes.

Remove from the source of heat and cool to room temperature.

Complete dissolution is not necessary. If any insoluble residue remains, filter the solution through a medium grade filter paper and wash thoroughly with water collecting the filtrate and washings in the volumetric flask.

Transfer to a 1 litre volumetric flask (5b)), dilute to the mark with water (4b)) and mix thoroughly.

7.2 Measurement of sodium and potassium

Operate the AAS in accordance with the manufacturer's instructions with the Na/K lamp in place. Whilst spraying water adjust the wave length setting around 589 nm (for Na) to give the maximum signal to the detector.

Use the burner height and fuel settings established in the calibration procedure. Adjust the gain control to give 0 % absorption whilst spraying the 0 mg/l calibration solution.

Spray one or more calibration solutions to confirm that the calibration graph is valid. If necessary prepare a new graph. Spray the sample solution and record the absorption. Refer to the calibration graph and record the concentration of the sample solution in mg Na_2O/litre.

If the absorption is off scale prepare a diluted extract by transferring 10,0 ml of the sample solution into a 100 ml volumetric flask and dilute to the mark with the 0 mg/l calibration solution i. e. dilution $D = 10$.

Mix thoroughly and repeat the procedure. Further dilutions may be required. Record mass m_2 of Na_2O in mg in 1 litre of solution.

Reset the wave length control to around 768 nm (for K) and repeat the above procedure. Record mass m_3 of K_2O in mg in 1 litre of solution.

7.3 Calculation of results

Calculate the Na_2O and K_2O content of the admixture from the following equations:

$$Na_2O \text{ content} = \frac{m_2}{m_1} \times 100\ D\ \%\ \text{by mass} \qquad \ldots (1)$$

$$K_2O \text{ content} = \frac{m_3}{m_1} \times 100\ D\ \%\ \text{by mass} \qquad \ldots (2)$$

Where m_1 = mass of admixture sample in mg and D = the dilution used if necessary.

Calculate the total equivalent alkali content of the admixture from the following equation:

$$\text{Total } Na_2O \text{ equivalent} = Na_2O \text{ content} + 0{,}658\ K_2O \text{ content}\ \%\ \text{by mass}$$

Report the total Na_2O equivalent content in percent per mass to the nearest 0,1 %.

NOTE: From 12 sets of results from different laboratories, after rejecting one set as outliers, the coefficient of variation was found to be 5 % of the mean for alkali contents in a range from 1,0 % to 3,5 % Na_2O equivalent.

8 Test report

a) Name or code of admixture tested (with information relating to its marking);

b) date of the test;

c) Name of the laboratory;

d) Name of operator;

e) Type of the equipment;

f) Origin of the sample and date when taken;

g) Sodium oxide content;

h) Potassium oxide content;

i) Sodium oxide equivalent content.