



SLOVENSKI STANDARD

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Admixture 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 Alkaligehalts von Zusatzstoffen

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

Ta slovenski standard je istoveten z: EN 480-12:2005

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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en

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EUROPEAN STANDARD
NORME EUROPÉENNE
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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, mortiers et coulis - Méthodes d'essai
- Partie 12: Détermination de la teneur en alcalis 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 28 July 2005.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. 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.

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.

CEN members are the national standards bodies of Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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Foreword

This European Standard (EN 480-12:2005) 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 2006, and conflicting national standards shall be withdrawn at the latest by April 2006.

It has been drafted by Subcommittee 3 (SC 3) of TC 104 "Admixtures for concrete, mortar and grout".

This document is part of the series EN 480 "Admixtures for concrete, mortar and grout – Test methods" which comprises 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 *Admixtures for concrete, mortar and grout - Test methods - Part 14: Measurement of corrosion susceptibility of reinforcing steel in concrete - Potentiostatic electro-chemical test method ¹⁾*

This document is applicable together with the other standards of the EN 480 series.

This document supersedes EN 480-12:1997.

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, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1) This part is under preparation

EN 480-12:2005 (E)**1 Scope**

This document 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

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 196-2, *Methods of testing cement – Determination of the chloride, carbon dioxide and alkali content of cement.*

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.

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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 document.

4 Reagents

4.1 Concentrated nitric acid, analytical reagent diluted 1: 1 with water (4.2).

4.2 Distilled, de-ionised or demineralised water. The same source of water shall be used for preparation of calibration solutions and sample extracts.

4.3 Sodium stock solution (100 mg Na₂O per litre). Dissolve 0,188 g sodium chloride, analytical reagent grade, dried at (105 ± 5) °C to constant mass, in water (4.2), dilute to 1 litre in a volumetric flask (5.2) with water²⁾.

4.4 Potassium stock solution (100 mg K₂O per litre). Dissolve 0,158 g potassium chloride, analytical reagent grade, dried at (105 ± 5) °C to constant mass, in water (4.2), dilute to 1 litre in a volumetric flask (5.2) with water²⁾.

4.5 Calibration solution³⁾. 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 (5.5).

Table 1: Calibration solutions

Na ₂ O and K ₂ O concentration	Sodium stock solution (4.3)	Potassium stock solution (4.4)	Nitric Acid (4.1)
mg/l	ml	ml	ml
0	0	0	10
2,5	25	25	10
5	50	50	10

2) Commercially available calibrated solutions for spectroscopic applications may be used.

3) The volumes of stock solutions given in Table 1 may not be appropriate when commercially available spectroscopic solutions are used. In addition some atomic absorption spectrophotometers may require a smaller range of Na₂O and K₂O concentration for calibration and use. In this case modify the calibration solutions accordingly.

EN 480-12:2005 (E)**5 Apparatus**

- 5.1** Balance with a capacity of up to 200 g readable to 0,1 mg.
- 5.2** 100 ml and 1 litre volumetric flasks of class A in accordance with ISO 1042.
- 5.3** Calibrated pipettes of class A in accordance with ISO 648.
- 5.4** 10 ml and 25 ml measuring cylinders.
- 5.5** 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.
- 5.6** 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.
- 5.7** A flame photometer (according to EN 196-2) if alternatively used in accordance with Clause 3.

6 Preparation of calibration graphs

Operate the AAS or the flame photometer 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 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 (5.2) with 150 to 250 ml water (4.2) 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 (4.2) and 10 ml nitric acid (4.1). 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 (5.2)), dilute to the mark with water (4.2)) 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 Na₂O/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 Na₂O 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 K₂O in mg in 1 litre of solution.

7.3 Results

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Calculate the Na₂O and K₂O content of the admixture from the following equations:

$$\text{Na}_2\text{O content} = \frac{m_2}{m_1} \times 100 D \text{ \% by mass} \quad (1)$$

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$$\text{K}_2\text{O content} = \frac{m_3}{m_1} \times 100 D \text{ \% by mass} \quad (2)$$

Where

m_1 mass of admixture sample in mg

m_2 mass of Na₂O in mg

m_3 mass of K₂O in mg

and

D the dilution used if necessary.

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

$$\text{Total Na}_2\text{O equivalent} = \text{Na}_2\text{O content} + 0,658 \text{ K}_2\text{O content} \text{ \% by mass}$$

Report the total Na₂O 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 % Na₂O equivalent.