# SLOVENSKI STANDARD
# kSIST FprEN 196-10:2015

**01-september-2015**

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**Metode preskušanja cementa - 10. del: Določevanje vodotopnega kroma (VI) v cementu**

Methods of testing cement - Part 10: Determination of the water-soluble chromium (VI) content of cement

Prüfverfahren für Zement - Teil 10: Bestimmung des Gehaltes an wasserlöslichem Chrom (VI) in Zement

**Ta slovenski standard je istoveten z:** **FprEN 196-10**

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| 91.100.10 | Cement. Mavec. Apno. Malta | Cement. Gypsum. Lime. Mortar |

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EUROPEAN STANDARD

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# FINAL DRAFT
# FprEN 196-10

June 2015

ICS 91.100.10

Will supersede EN 196-10:2006

English Version

# Methods of testing cement - Part 10: Determination of the water-soluble chromium (VI) content of cement

Prüfverfahren für Zement - Teil 10: Bestimmung des Gehaltes an wasserlöslichem Chrom (VI) in Zement

This draft European Standard is submitted to CEN members for unique acceptance procedure. It has been drawn up by the Technical Committee CEN/TC 51.

If this draft becomes a European Standard, 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.

This draft European Standard was established by CEN 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 CEN-CENELEC Management Centre has the same status as the official versions.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

**CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels**

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# Foreword

This document (FprEN 196-10:2015) has been prepared by Technical Committee CEN/TC 51 “Cement and building limes”, the secretariat of which is held by IBN.

This document is currently submitted to the Unique Acceptance Procedure.

This document supersedes EN 196-10:2006.

In comparison to EN 196-10:2006, the following changes have been made:

— In Clause 2, the normative references have been updated;

— The standard has been editorially revised.

EN 196 consists of the following parts, under the general title *Methods of testing cement:*

— *Part 1: Determination of strength;*

— *Part 2: Chemical analysis of cement;*

— *Part 3: Determination of setting times and soundness;*

— *Part 4: Quantitative determination of constituents* (CEN/TR 196-4) ;

— *Part 5: Pozzolanicity test for pozzolanic cement;*

— *Part 6: Determination of fineness;*

— *Part 7: Methods of taking and preparing samples of cement;*

— *Part 8: Heat of hydration - Solution method;*

— *Part 9: Heat of hydration - Semi-adiabatic method;*

— *Part 10: Determination of the water-soluble chromium (VI) content of cement.*

# Introduction

This European Standard specifies the reference method for the determination of water-soluble chromium (VI) content of cement that consists of two stages, an extraction procedure and an analysis of the filtered extract.

This European Standard test method has adopted the principle that extraction is carried out under conditions approximating as closely as possible to those during the commercial use of cement. Consequently extraction is by standard mortar and subsequent filtration. Other extraction procedures based on paste extraction have traditionally been used and are included in Annexes C and D for use as screening tests, in factory production control or laboratories not having access to equipment specified in FprEN 196-1 for the production of mortar. The use of paste extraction is outside the normal conditions of use of cement.

This European Standard test method has adopted the principle of analysis by spectrophotometry. The procedures set down generally permit the analysis to be carried out without the need for an oxidation step. On rare occasions some cements may contain reducing species, not controlled by the routine method, that interfere with the analysis and require an oxidation step. Inter-laboratory testing has demonstrated that it is necessary to include an ‘initial assessment test’ in order to observe the effects on the analysis. By comparing the results obtained from the method with and without the oxidation step, it can be determined whether, for that cement, the reference method should include the oxidation step.

Other instrumental procedures may be used for the analysis of the filtered extract provided they are calibrated against the analysis of the filtered extract using the reference procedure.

In case of dispute or failure to comply with a regulatory limit only the reference method shall be used.

This European Standard test method has drawn heavily on the Danish Standard DS 1020 and the extraction procedure developed by the French cement industry association ATILH. Careful consideration has been given to the details of the German TRGS 613 method developed by Germany’s Hazardous Materials Committee in support of Industrial Regulations for Hazardous Materials. Notice was also taken of the British Cement Association ‘inherent colour’ method; the draft method produced by CEN/TC 193/WG1, reference N680, for cement-based adhesives; European Standard method EN 420 for protective gloves; and to the method, reference ID-215, developed by the Occupational Safety and Health Administration, Salt Lake City, UT, USA.

The USA Portland Cement Association, Research and Development report Serial No. 2554 “Review and evaluation of analytical methods for the determination of hexavalent chromium in hydraulic cements and clinker” by Waldemar A. Klemm was found to be most helpful in resolving technical issues. CEN/TR 14589 confirmed that chromium species and solubilities are sensitive to pH and redox conditions and care has been taken to address these in this European Standard by controlling sample exposure to air, by adding the indicator to the alkaline filtered extract and by precisely specifying the pH for the analytical procedure.

This European Standard test method was developed in order to provide a reference test method for use in the evaluation of compliance of cement with the requirements in entry 47 to Annex XVII of Regulation (EC) 1907/2006 of the European Parliament and of the Council of 18 December 2006 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH). A system for the evaluation of compliance of cement is set out in Annex A.

# 1 Scope

This part of EN 196 specifies the method for the determination of the water-soluble chromium (VI) content of cement.

A reference method is described consisting of two stages, an extraction procedure and an analysis of the filtered extract. Guidance on other extraction procedures, suitable for screening tests, for factory production control or other purposes, is given but in case of dispute or failure to comply with a regulatory limit only the reference method is used. The reference method has alternatives whereby the filtered extract may be subjected to an oxidation step or not. The criteria by which the appropriate procedure is selected are set down. Other instrumental procedures may be used for the analysis of the filtered extract provided they are calibrated against the analysis of the filtered extract using the reference procedure. In the case of a dispute, only the reference method is used.

Annex A sets out a normative procedure to be followed in case this test method is used as the basis for evaluation of conformity of a cement with the regulatory limit in Regulation (EC) No. 1907/2006.

This part of EN 196 describes a method that applies to cements. It may have wider applicability but this would need to be verified by testing on a product-by-product basis. Guidance in the possible application of this European Standard to the determination of the water-soluble chromium (VI) content of cement-containing preparations is given in Annex B.

Annexes C and D provide information on other test procedures based on paste extraction and thus depart from the performance of cement in its normal conditions of use. They may be carried out with or without the oxidation process. Users should be aware that results using these methods might be significantly different to those obtained by the reference method. In the case of dispute or failure to comply with the regulatory limit only the reference method is used.

Annex E provides guidance on a method for determination of the excess reducing agent content of cement as used in the factory internal control system of some countries. Manufacturers using such an internal control method should assure themselves of the relevance of results in comparison with testing by the reference method.

# 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 196-7, *Methods of testing cement - Part 7: Methods of taking and preparing samples of cement*

EN ISO/IEC 17020, *Conformity assessment - Requirements for the operation of various types of bodies performing inspection (ISO/IEC 17020)*

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025)*

EN ISO/IEC 17065, *Conformity assessment - Requirements for bodies certifying products, processes and services (ISO/IEC 17065)*

# 3 General requirements for testing

## 3.1 Number of tests

Determination of water-soluble chromium (VI) content of cement, where the determination is not part of a series subject to statistical control, shall be carried out in duplicate.

Where the determination is one of a series subject to statistical control, a single test shall be the minimum required.

In the case of a dispute, the number of tests shall be two (see also 3.3).

## 3.2 Repeatability and reproducibility

Repeatability - precision under repeatability conditions where independent test results are obtained with the same method on identical test items (material) in the same laboratory by the same operator using the same equipment within short intervals of time.

Reproducibility - precision under reproducibility conditions where test results are obtained with the same method on identical test items (material) in different laboratories with different operators using different equipment.

Repeatability and reproducibility in this European Standard are expressed as repeatability standard deviation and reproducibility standard deviation in percent by mass.

## 3.3 Expression of masses, volumes, factors and results

Express masses in the extraction stage in grams to the nearest 0,1 g. Express masses in the analytical stage in grams to the nearest 0,000 1 g and volumes from burettes in millilitres to the nearest 0,05 ml unless otherwise specified.

Express the calculated results, where a single test result has been obtained, as a percentage of the cement as received.

Express the calculated results, where two test results have been obtained, as the mean of the results, as a percentage of the cement as received.

If the two test results differ by more than twice the standard deviation of repeatability, repeat the test and take the mean of the two closest test results.

Express the reported water-soluble chromium (VI) content as a percentage of the cement as received, to four decimal places.

Where the results of determinations with and without oxidation are to be compared results shall be considered equivalent if they do not differ by more than twice the standard deviation of repeatability.

The results of all individual tests shall be recorded.

## 3.4 Blank determinations

Carry out a blank determination without a sample following the same procedure and using the same amounts of reagents. Correct the results obtained for the analytical determination accordingly.