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



EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

		This Estonian standard EVS-EN 196-10:2016 consists of the English text of the European standard EN 196-10:2016.
Standard on jõustunud sellekoh avaldamisega EVS Teatajas.	ase teate	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 18.05.2016.		Date of Availability of the European standard is 18.05.2016.
Standard on kättesaadav Standardikeskusest.		The standard is available from the Estonian Centre for Standardisation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile <u>standardiosakond@evs.ee</u>.

ICS 91.100.10

Standardite reprodutseerimise ja levitamise õigus kuulub Eesti Standardikeskusele

Andmete paljundamine, taastekitamine, kopeerimine, salvestamine elektroonsesse süsteemi või edastamine ükskõik millises vormis või millisel teel ilma Eesti Standardikeskuse kirjaliku loata on keelatud.

Kui Teil on küsimusi standardite autorikaitse kohta, võtke palun ühendust Eesti Standardikeskusega: Aru 10, 10317 Tallinn, Eesti; koduleht <u>www.evs.ee</u>; telefon 605 5050; e-post <u>info@evs.ee</u>

The right to reproduce and distribute standards belongs to the Estonian Centre for Standardisation

No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying, without a written permission from the Estonian Centre for Standardisation.

If you have any questions about copyright, please contact Estonian Centre for Standardisation:

Aru 10, 10317 Tallinn, Estonia; homepage www.evs.ee; phone +372 605 5050; e-mail info@evs.ee

EUROPEAN STANDARD NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 196-10

May 2016

ICS 91.100.10

Supersedes EN 196-10:2006

English Version

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

Méthodes d'essais des ciments - Partie 10 : Détermination de la teneur en chrome (VI) soluble dans l'eau des ciments Prüfverfahren für Zement - Teil 10: Bestimmung des Gehaltes an wasserlöslichem Chrom (VI) in Zement

This European Standard was approved by CEN on 20 December 2015.

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 CEN-CENELEC Management Centre 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 CEN-CENELEC Management Centre has the same status as the official versions.

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



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

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

Conte	ents	Page
Europe	ean foreword	3
- Introd	uction	4
1	Scope	5
2	Normative references	5
3 3.1 3.2 3.3 3.4	General requirements for testing Number of tests Repeatability and reproducibility Expression of masses, volumes, factors and results Blank determinations	6 6 6
4	Reagents	6
5	Apparatus	7
6	Preparation of a test sample of cement	8
7 7.1 7.2 7.3	Extraction procedure Principle Preparation of mortar Filtration	9 9 10
8 8.1 8.2 8.3	Determination of chromium (VI)	10 10
9 9.1 9.2	Calculation and expression of results Expression of results	12 13
10	Reporting of results	
11	Repeatability and reproducibility	13
Annex	A (normative) Evaluation of the compliance of cement with the regulatory limit in 47 to Annex XVII of Regulation (EC) No. 1907/2006 on water-soluble hexavalent chromium content	14
Annex	B (informative) Guidance on the application of this European Standard to the determination of the water soluble chromium (VI) content of cement-containing preparations	24
Annex	C (informative) Guidance on screening test method (1) using paste extraction - method based on DS 1020	26
Annex	D (informative) Guidance on screening test method (2) using paste extraction - method based on TRGS 613	29
Annex	E (informative) Guidance on the photometric determination of chromium reduction capacity in cements	33
Bibliog	graphy	37

European foreword

This document (EN 196-10:2016) has been prepared by Technical Committee CEN/TC 51 "Cement and building limes", the secretariat of which is held by NBN.

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 November 2016, and conflicting national standards shall be withdrawn at the latest by November 2016.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

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.

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

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 EN 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) 1907/2006¹ 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.

4

 $^{^1}$ Note that Regulation (EC) No 1907/2006 is impacted by Commission Regulation (EU) No 126/2013 of 13 February 2013 amending Annex XVII to Regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH).

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) $1907/2006^2$.

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 ensure 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)

² Note that Regulation (EC) No 1907/2006 is impacted by Commission Regulation (EU) No 126/2013 of 13 February 2013 amending Annex XVII to Regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH).