



Water quality – Determination of chromium - Atomic absorption spectrometric method

The European Standard EN 1233:1996 has the status of a Swedish Standard. This document contains the official English version of EN 1233:1996.

This standard specifies two methods for the determination of chromium in water.

- flame atomic absorption spectrometry

Applicable to the analysis of water and waste water when the concentration range is between 0,5 mg/l and 20 mg/l.

- electrothermal atomization atomic absorption spectrometry

Applicable to the analysis of water and waste water when the concentration range is between 5 µg/l and 100 µg/l. Higher concentrations can be determined by using a smaller sample volume.

This standard supersedes the parts concerning determination of chromium in the standards SS 02 81 52 and SS 02 81 84.

Vattenundersökningar - Bestämning av krom med atomabsorptionsspektrometri

Europastandarden EN 1233:1996 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN 1233:1996.

Standarden beskriver två metoder för bestämning av krom i vatten:

- atomabsorptionsspektrometri i flamma

Lämplig för bestämning av krom i vatten och avlopp med en kromkoncentration mellan 0,5 mg/l och 20 mg/l.

- atomabsorptionsspektrometri med grafitugn (elektrotermisk atomisering)

Lämplig för analys av krom i vatten och avlopp med en kromkoncentration mellan 5 µg/l och 100 µg/l. Högre koncentrationer går att bestämma genom att använda en mindre provvolym.

Standarden ersätter avsnitten som behandlar bestämning av krom i standarderna SS 02 81 52 respektive SS 02 81 84.

EUROPEAN STANDARD
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EUROPÄISCHE NORM

EN 1233

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Descriptors: water, quality, chemical analysis, determination of content, chromium, atomic absorption spectrometry

English version

**Water quality - Determination of chromium -
Atomic absorption spectrometric method**

Qualité de l'eau – Dosage du chrome –
Méthodes par spectrométrie d'absorption
atomique

Wasserbeschaffenheit – Bestimmung von
Chrom – Verfahren mittels
Atomabsorptionsspektrometrie

This European Standard was approved by CEN on 1996-04-19. 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.

The European Standards exist 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, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

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

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

Foreword

This European Standard has been prepared by the Technical Committee CEN/TC 230 "Water analysis", the secretariat of which is held by DIN.

Annex A is informative.

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 January 1997, and conflicting national standards shall be withdrawn at the latest by January 1997.

In accordance 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, United Kingdom.

On the elaboration of this European Standard, the revision of the International Standard ISO 9174 has been started simultaneously with the intention to harmonize both standards.

Introduction

Chromium occurs in water in the oxidation states III and VI. The two methods described determine chromium in both oxidation states, either as acid soluble chromium or as water soluble chromium, depending on the sample pretreatment. The method chosen depends on the concentration of chromium in the water to be examined. Some information is given in an informative annex on pretreatment and digestion of chromium from sludges and sediments.

1 Scope

This European Standard specifies two methods for the determination of chromium in water by atomic absorption spectrometry. The two methods are covered in separate clauses as follows:

- Clause 3: Determination of chromium by flame atomic absorption spectrometry;
- Clause 4: Determination of chromium by electrothermal atomization atomic absorption spectrometry.

Clause 3 is applicable to the analysis of water and waste water when the concentration range is between 0,5 mg/l and 20 mg/l of chromium. When the concentration is below 0,5 mg/l the determination can be carried out after carefully evaporating an acidified sample to small volume, taking care to avoid the formation of a precipitate.

WARNING: The use of evaporation will increase the effect of interfering substances and therefore for concentrations below 0,1 mg/l the method in clause 4 is given.

Clause 4 is applicable to the analysis of water and waste water when the concentration range is between 5 µg/l and 100 µg/l of chromium by injecting a sample volume of 20 µl. It is applicable to the determination of higher concentrations by using a smaller sample volume.

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 amendment to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

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| EN 25667-2:1993 | Water quality - Sampling - Part 2: Guidance on sampling techniques (ISO 5667-2: 1991). |
| EN ISO 5667-3:1995 | Water quality - Sampling - Part 3: Guidance on the preservation and handling of samples (ISO 5667-3: 1994). |

3 Determination of chromium by flame atomic absorption spectrometry

3.1 Principle

The method is based on the atomic absorption spectrometric measurement of the chromium content of the acidified sample in a nitrous oxide/acetylene flame. Measurement at a wavelength of 357,9 nm. Addition of lanthanum salt to reduce matrix interferences is necessary.

3.2 Reagents

3.2.1 General

All reagents shall be of recognized analytical grade. Use deionized water or water distilled from an all glass apparatus. The water used for blank tests and for preparing reagents and standard solutions shall have a chromium content that is negligible compared with the smallest concentrations to be determined in the samples.

3.2.2 Hydrochloric acid, HCl, $\rho \approx 1,18$ g/ml.

3.2.3 Nitric acid, HNO₃, $\rho \approx 1,42$ g/ml.

3.2.4 Nitric acid, $c(\text{HNO}_3) = 1,5 \text{ mol/l}$.

Introduce about 500 ml of water into a 1000 ml volumetric flask, add 100 ml of nitric acid (see 3.2.3) and dilute to the mark with water.

3.2.5 Hydrogen peroxide, H_2O_2 , 30 % (m/m) solution,

3.2.6 Lanthanum chloride, (LaCl_3) , solution with $\rho(\text{La}) = 20 \text{ g/l}$.

Dissolve 23,5 g of lanthanum oxide La_2O_3 in 200 ml of hydrochloric acid (see 3.2.2), dilute to 1000 ml with water and mix.

Appropriate precautions shall be observed when preparing this solution because the reaction of La_2O_3 with hydrochloric acid is strongly exothermic.

3.2.7 Chromium solutions

3.2.7.1 Chromium, stock solution, with $\rho(\text{Cr}) = 1,000 \text{ g/l}$.

Dry a portion of potassium bichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) at $105 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ for about 2 h. Cool and dissolve $2,825 \text{ g} \pm 0,001 \text{ g}$ of the dried potassium bichromate in water. Add $5 \text{ ml} \pm 1 \text{ ml}$ of nitric acid (see 3.2.3) and dilute to 1000 ml with water in a volumetric flask.

1 ml of this stock solution contains 1,00 mg of chromium.

Store this solution in either polyethylene or borosilicate glass containers at room temperature. The solution is stable at room temperature for about 1 year if it is stored in the dark and at a pH between 1 and 2.

NOTE: Chromium stock solutions are commercially available.

3.2.7.2 Chromium, standard solution with $\rho(\text{Cr}) = 50 \text{ mg/l}$.

Introduce $50,00 \text{ ml} \pm 0,01 \text{ ml}$ of the chromium stock solution (see 3.2.7.1) into a 1000 ml volumetric flask. Add 1 ml of nitric acid (see 3.2.3), make up to the mark with water and mix.

This solution is stable for at least 1 month,

3.3 Apparatus

3.3.1 Atomic absorption spectrometer, equipped with a chromium hollow cathode lamp and a nitrous oxide/acetylene burner, and operated in accordance with the manufacturer's instructions. It is essential that the manufacturer's safety recommendations are strictly observed when using the nitrous oxide/acetylene flame.

3.3.2 Glassware

Before use carefully soak all glassware for about 24 h in nitric acid (see 3.2.4), then rinse thoroughly with water.

If low concentrations are expected (for example in ground water) the glassware should be kept under nitric acid (see 3.2.4) until use.

Do not use glassware which has been cleaned with chromic acid.

3.3.3 Membrane filters, of nominal pore diameter $0,45 \text{ } \mu\text{m}$, washed thoroughly with nitric acid (see 3.2.4) and rinsed with water (see 3.2.1).

3.4 Sampling and preparation of test portions

3.4.1 General

Collect and preserve samples according to EN 25667-2 and EN ISO 5667-3. See also annex A .
Collect samples in high density polyethylene or borosilicate glass containers which have been previously cleaned with nitric acid (see 3.2.4) and then rinsed with water (see 3.2.1).