



SLOVENSKI STANDARD
SIST EN 1233:1997

01-avgust-1997

Kakovost vode - Določanje kroma - Atomska absorpcijska spektrofotometrijska metoda

Water quality - Determination of chromium - Atomic absorption spectrometric methods

Wasserbeschaffenheit - Bestimmung von Chrom - Verfahren mittels Atomabsorptionsspektrometrie

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

iTeh STANDARD PREVIEW
(standards.itih.ai)

[SIST EN 1233:1997](https://standards.itih.ai/catalog/standards/sist/129cc120-08d0-4902-985a-6cd5da8656e5/sist-en-1233-1997)

Ta slovenski standard je istoveten z: EN 1233:1996

<https://standards.itih.ai/catalog/standards/sist/129cc120-08d0-4902-985a-6cd5da8656e5/sist-en-1233-1997>

ICS:

13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances
-----------	---------------------------------	--

SIST EN 1233:1997

en

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST EN 1233:1997

<https://standards.iteh.ai/catalog/standards/sist/129cc120-08d0-4902-985a-ded3da8656e5/sist-en-1233-1997>

EUROPEAN STANDARD

EN 1233

NORME EUROPÉENNE

EUROPÄISCHE NORM

July 1996

ICS 13.060.40

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

English version

**Water quality - Determination of chromium -
Atomic absorption spectrometric methods**Qualité de l'eau - Dosage du chrome - Méthodes
par spectrométrie d'absorption atomiqueWasserbeschaffenheit - Bestimmung von Chrom -
Verfahren mittels Atomabsorptionsspektrometrie**(standards.iteh.ai)**

SIST EN 1233:1997

<https://standards.iteh.ai/catalog/standards/sist/129cc120-08d0-4902-985a-ded3da8656e5/sist-en-1233-1997>

This European Standard was approved by CEN on 1996-05-16. 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.

CENEuropean 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.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST EN 1233:1997

<https://standards.iteh.ai/catalog/standards/sist/129cc120-08d0-4902-985a-ded3da8656e5/sist-en-1233-1997>

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.

- 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 dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) at $105 \text{ °C} \pm 2 \text{ °C}$ for about 2 h. Cool and dissolve 2,825 g $\pm 0,001 \text{ g}$ of the dried potassium dichromate in water. Add 5 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 polyethene 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 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.

SIST EN 1233:1997

3.3 Apparatus <https://standards.iteh.ai/catalog/standards/sist/129cc120-08d0-4902-985a-ded3da8656e5/sist-en-1233-1997>

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 μm , 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 polyethene 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).

3.4.2 Acid soluble Chromium

Treat the samples by addition of sufficient nitric acid (see 3.2.3), immediately after collection, to adjust the pH to between 1 and 2.

To 90 ml of the acidified sample, or another suitable aliquot in accordance with the expected concentration, add 1 ml of hydrogen peroxide (see 3.2.5) and 2 ml of nitric acid (see 3.2.3). Boil and evaporate to a volume of approximately 50 ml.

The sample shall not be reduced to dryness.

In the case of samples with low chromium concentrations this digestion method should be carried out in an autoclave or a microwave oven.

It is important to follow the manufacturer's instructions.

Add 10 ml of nitric acid (see 3.2.3) to the evaporated solution. Transfer the solution to a 100 ml volumetric flask. Add 10 ml of lanthanum chloride solution (see 3.2.6) into the flask, dilute to the mark with water and mix.

3.4.3 Water Soluble chromium

Filter the sample through a membrane filter (see 3.3.3), as soon as possible after collection, and acidify the filtrate immediately with nitric acid (see 3.2.3) to give a pH between 1 and 2.

Add 10 ml of lanthanum chloride solution (see 3.2.6) to a 100 ml volumetric flask and make up to the mark with the acidified filtrate, or use another suitable aliquot in accordance with the expected concentration, and mix.

3.5 Procedure

3.5.1 Blank test

Carry out a blank test in parallel with the determination by the same procedure and using the same quantities of all the reagents as in the sampling and determination, but replacing the test portion by water.

3.5.2 Preparation of the calibration solutions

Before each set of determinations, prepare from the chromium standard solution (see 3.2.7.2) at least five calibration solutions covering the range of concentrations to be determined; for example: pipette 1,0 ml, 2,5 ml, 5,0 ml, 10,0 ml and 20,0 ml of chromium standard solution (see 3.2.7.2) into a series of 100 ml volumetric flasks. Add to each flask depending on the pretreatment (see 3.4.2 and 3.4.3) 10 ml or 2 ml of nitric acid (see 3.2.3) respectively and 10 ml of lanthanum chloride solution (see 3.2.6), dilute to the mark with water and mix. These solutions correspond to chromium concentrations of 0,50 mg/l; 1,25 mg/l; 2,50 mg/l; 5,00 mg/l and 10,0 mg/l respectively. Proceed accordingly for other concentration ranges of interest.

3.5.3 Calibration

Set up the instrument in accordance to the manufacturer's instructions (at wavelength $\lambda = 357,9$ nm) using a nitrous oxide/acetylene flame.

Aspirate a calibration solution (see 3.5.2) and optimize the aspiration, the burner height and the flame conditions. Adjust the response of the instrument to zero absorbance with water.

In turn, aspirate the set of calibration solutions (see 3.5.2) and the blank solution (see 3.5.1). Plot a graph having the chromium concentrations of the calibration solutions in milligrams per litre as abscissa and the corresponding absorbance values as ordinate. Alternatively, data processing facilities can be used to equate the concentration against the absorbance.

3.5.4 Test portion measurement

Aspirate the prepared test portion (see 3.4.2 or 3.4.3) into the flame and measure the absorbance for chromium. After each measurement aspirate water and readjust the zero, if necessary.

3.6 Expression of results

3.6.1 By reference to the calibration graph obtained (see 3.5.3) or data processing facilities used, determine the concentration of chromium corresponding to the absorbances of the test portion (see 3.5.4) and of the blank solution (see 3.5.1).

Calculate the concentration of chromium of the sample using the equation

$$\rho = \frac{(A_s - A_{s0}) \cdot V_w}{V_p \cdot b}$$

where:

- ρ is the chromium concentration of the sample, in milligrams per litre;
- A_s is the absorbance of the test portion;
- A_{s0} is the absorbance of the blank;
- V_p is the volume in ml of the acidified sample in accordance with 3.4.2 or 3.4.3, respectively, in millilitres;
- b is the sensitivity (slope of the calibration graph) in litre per milligrams;
- V_w is the volume of the test portion, according to 3.4.2 respectively 3.4.3, in millilitres.

3.6.2 Precision data

An interlaboratory test carried out in January 1985 yielded the data given in table 1:

Table 1: Precision data for clause 3

Type of sample	l	n	x_i	\bar{X}	y	σ_r	VC_r	σ_R	VC_R
Standard solution	11	43	15,0	14,64	97,5	0,084	0,6	0,798	5,5
Water with added concentration	14	56	2,0	2,06	103,0	0,053	2,6	0,218	10,6

where:

- l is the number of laboratories;
- n is the number of measured values (without outliers);
- x_i is the concentration of samples, in milligrams per litre;
- \bar{X} is the overall mean, in milligrams per litre;
- y is the recovery rate, in percent;
- σ_r is the repeatability standard deviation, in milligrams per litre;
- VC_r is the repeatability variation coefficient, in percent;
- σ_R is the reproducibility standard deviation, in milligrams per litre;
- VC_R is the reproducibility variation coefficient, in percent.

The standard solution used was made from deionized water, spiked with chromium. The water used was a municipal, biologically treated waste water, spiked with chromium. The digestion as described in 3.4.2 was applied; in the atomic absorption spectrometer apparatus, a hollow cathode lamp was used.

3.7 Interferences

The following ions have been tested and shown not to cause interference below the concentration given in table 2.

This information was taken from practical tests carried out in a working group in 1984.

Table 2: Interfering ions

Ion	Concentration, mg/l	Ion	Concentration mg/l
Sulfate	10 000	Iron	500
Chloride	12 000	Nickel	100
Sodium	9 000	Copper	100
Potassium	9 000	Cobalt	100
Magnesium	2 000	Aluminium	100
Calcium	2 000	Zinc	100

The total dry residue of the sample shall not exceed 15 g/l and the conductivity should be lower than 2000 mS/m.

NOTE: It is possible to determine the effect of possible interferences by analysing samples spiked with chromium and various concentrations of the potential interfering substance using the method of standard additions.

3.8 Test report

The test report shall contain at least the following information:

- a) a reference to this European Standard;
- b) a reference to the procedure used;
- c) complete identification of the sample;
- d) any other information relevant to the procedure.

4 Determination of chromium by electrothermal atomization atomic absorption spectrometry

4.1 Principle

Chromium is determined directly in acidified samples by electrothermal atomization atomic absorption spectrometry. The sample is introduced into an electrically heated graphite tube and the absorbance is measured at a wavelength of 357,9 nm. If necessary, the standard additions technique is employed.

4.2 Reagents

4.2.1 See 3.2.

4.2.2 Chromium standard solution, with $\rho(\text{Cr}) = 0,5 \text{ mg/l}$

Add 10,00 ml \pm 0,01 ml of the chromium standard solution (see 3.2.7.2) to a 1000 ml volumetric flask. Add 10 ml of nitric acid (see 3.2.3) and dilute to the mark with water.

This solution is stable for at least 1 month.

4.3 Apparatus

4.3.1 Atomic absorption spectrometer, equipped with an electrothermal atomizer, chromium hollow cathode lamp and a suitable device for correction of nonspecific absorbants.

4.3.2 Atomizer tubes

The atomizer tubes will vary according to the instrument used but shall be pyrolytically coated and pre-conditioned before use in accordance with the manufacturer's instructions.

NOTE: It is permissible to use a L'vov platform.

4.3.3 Automatic sample injector (autosampler) or micro-syringe, of appropriate volume

For high precision an automatic sample injector is necessary.

4.4 Sampling and preparation of test portions

Follow the procedures described in 3.4 but omit the lanthanum chloride solution.

Because of the very low levels of chromium being determined, take particular care to avoid any contamination at all stages of sampling and analysis.