

SLOVENSKI STANDARD oSIST prEN ISO 18724:2024

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Kakovost vode - Določanje kroma (IV), raztopljenega v vodi - Fotometrijska metoda (ISO/DIS 18724:2024)

Water quality - Determination of dissolved chromium(VI) in water - Photometric method (ISO/DIS 18724:2024)

Wasserbeschaffenheit - Bestimmung des gelösten Chrom(VI) in Wasser - Photometrisches Verfahren (ISO/DIS 18724:2024)

Qualité de l'eau - Détermination du chrome dissous(VI) dans l'eau - Méthode photométrique (ISO/DIS 18724:2024)

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Water quality — Determination of dissolved chromium(VI) in water — Photometric method

Qualité de l'eau — Détermination du chrome dissous(VI) dans l'eau — Méthode photométrique

ICS: 13.060.50

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

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Introduction

Chromium is an important raw material in the industrial manufacturing process that can contaminate sources of drinking water (e.g. groundwater, surface water). The most common oxidation states of chromium are +3 and +6. The hexavalent form (Cr(VI)) is far more toxic and water-polluting than the trivalent form (Cr(III)).

The chromium content depends on several factors: the availability of chromium in the rock, the weathering stage and the vicinity. Chromium(VI) occurs mainly in the aerobic environment; under reducing / anaerobic conditions, chromium(VI) is reduced to chromium(III).

Chromium(VI) occurs naturally in groundwater aquifers, mostly in low concentrations depending on the occurrence in geological formations, e.g., volcanic rocks.

Other sources of chromium(VI) contamination for the environment are industrial activities, e.g., the production of textiles, leather tanning or electroplating.

A sensitive and specific photometric method for the determination of hexavalent chromium is the reaction with 1,5-diphenylcarbazide (DPC). Most of the standardized procedures are based on DPC chemistry, specifying different reaction conditions.

This procedure describes a uniform procedure that can be used for different photometric measuring devices, such as static or dynamic techniques. The available techniques vary in sensitivity. The choice of the technique used for the measurement depends on the chromium(VI) concentration expected in the sample.

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Water quality — Determination of dissolved chromium(VI) in water — Photometric method

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

1 Scope

This document specifies a method for the photometric determination of dissolved chromium(VI) using manual, e.g., hand photometry, or automated static, e.g., discrete analyser system, or automated dynamic techniques, e.g., flow injection analysis (FIA), continuous flow analysis (CFA), or ion chromatography with post-column derivatization (IC-PCR).

The choice of the analytical technique to be used and needs-based sample preparation (e.g., matrix elimination) enables the determination of chromium(VI) in concentrations $\geq 0,02 \ \mu g/l$ in raw water, drinking water, surface water, aqueous eluates, cooling water and treated wastewater, provided that the Matrix does not contain any reducing substances. Typical areas of application for the static techniques as well as FIA and CFA are samples with chromium(VI) concentrations $\geq 2 \ \mu g/l$. When using cuvettes with large optical path lengths, e.g. >100 mm, the range of application can be extended to concentrations <2 $\ \mu g/l$ chromium(VI) (Annex A; Annex B; Annex C, Clauses C.1 and C.2). When using coupled techniques (e.g. IC-PCR), chromium(VI) concentrations $\geq 0,02 \ \mu g/l$ can be determined (Annex C, Clause C.3).

In some waters with reducing ingredients, chromium(VI) losses can occur just a few hours after sampling. Therefore, the method was not validated for leachate from landfills and raw sewage.

2 Normative references OSIST prEN ISO 18724:20

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 7393-2, Water quality — Determination of free chlorine and total chlorine — Part 2: Colorimetric method using N,N-dialkyl-1,4-phenylenediamine, for routine control purposes

ISO 8466-1, Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function

ISO 8466-2, Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions

ISO/TS 13530, Water quality — Guidance on analytical quality control for chemical and physicochemical water analysis

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at <u>https://www.iso.org/obp</u>

IEC Electropedia: available at <u>https://www.electropedia.org/</u>

4 Interferences

4.1 General

The influence of the matrix interferences can vary considerably and depends on the type of sample. Matrix interferences can be detected by recovery or spiking attempts.

4.2 Sampling, preservation, storage

Oxidizing agents (e.g., peroxyacetic acid, permanganate) or chlorine, chlorine dioxide, hydrogen peroxide, and ozone used for disinfection in drinking water treatment can interfere. If necessary, their presence can be checked according to ISO 7393-2 or with potassium iodine starch test paper ($\underline{6.21}$) and eliminated by adding sodium sulfite ($\underline{6.18}$ or $\underline{6.19}$). Excess sulfite is removed with sodium hypochlorite solution II ($\underline{6.15}$).

NOTE 1 The oxidizing agents listed in <u>Table 1</u> do not interfere if the concentrations given are not exceeded.

Table 1 — Upper limit^[6][7] for mass concentrations of individual disinfectants up to which no significant interference occurs under the given reaction conditions

Disinfectant	Concentration
	in mg/l
chlorine	0,6
chlorine dioxide	0,4
hydrogen peroxide	0,2
ozone 11em Sta	undards 0,1

Sulfide in concentrations >0,2 mg/l interferes with the method.

Changes in valence are prevented by adding ammonium hydroxide/ammonium sulfate solution ($\underline{6.7}$) at a resulting pH between 8 and 9,5 (<u>Clause 8</u>).

NOTE 2 Slow reduction of chromium(VI) may occur in some waters.

Acidic preservation of the samples should be avoided because of the increased oxidation potential of (1) chromium(VI) in acids and the associated reduction of chromium(VI)^[1],^[2].

Chromium(III) and other metal ions present in the sample can be removed at the sampling point using a cation exchange cartridge $(\underline{7.7}, \underline{[3]})$ or precipitated in ammonium hydroxide/ammonium sulfate solution (<u>6.7</u>) with aluminium sulfate solution (<u>6.17</u>) as a precipitation aid and removed by filtration.

4.3 Interferences in photometry

Interferences caused by the sample's own colour shall be taken into account (8.3).

Samples with a pH value >9,5 shall be adjusted to a pH value between 8 and 9,5 by adding sulfuric acid (6.3) before analysis in order to comply with the requirements of 9.2.

Vanadium ions interfere when their concentration is ten times higher than that of chromium(VI) and generally in concentrations >4 mg/l.

Molybdenum and mercury ions react with the reagent to form a red dye. The intensity of the dye is significantly lower than that formed with chromium(VI), so that concentrations of up to 200 mg/l molybdenum and mercury can be tolerated at the adjusted pH value. However, such concentrations are extremely unlikely in the aqueous matrices covered by this document.

Iron in concentrations >1 mg/l can produce a yellow colour, but this is not a significant interference at the specified wavelength. Iron in particulate form should be removed by filtration.

NOTE See also ISO 11083:1994^[2].

5 Principle

Chromium(VI) reacts with 1,5-diphenylcarbazide to form a red-violet chromium-1,5-diphenylcarbazone complex. The absorbance of this complex is measured at (540 ± 10) nm (absorbance maximum at 544 nm). The reaction can optionally be carried out using manual (e.g., classical photometry) or automated static (e.g., single analysis system) or automated dynamic techniques (e.g., flow injection analysis technique (FIA), continuous flow analysis technique (CFA) or ion chromatography (IC)). <u>Annex A</u> provides information on manual static techniques, <u>Annex B</u> on automated static techniques, and <u>Annex C</u> describes dynamic techniques.

The chromium(VI) concentration is determined by calibration according to ISO 8466-1 or ISO 8466-2.

To check the validity of the calibration function determinations can be necessary. The method of standard addition can be applied if matrix interferences are expected.

Control experiments are necessary to ensure the validity of the calibration function. Replicate determinations may be necessary. The method of standard addition can be applied if matrix interferences are expected.

The user may choose other measurement techniques than those described in <u>Annexes A</u>, <u>B</u> and <u>C</u> as long as no changes are made to the sampling and pre-treatment (<u>Clause 8</u>) or to the required reagent concentrations in the reaction vessel (<u>9.2</u>, <u>9.3</u>).

6 Reagents

6.1 General

If available, reagents of quality "for analysis" (or better quality) that are free of chromium(VI) compounds are to be used. Carry out weighing with an accuracy of 1 % of the nominal mass, unless stated otherwise. If necessary, alternative concentrations and volumes to the solutions described below may be prepared and used. Alternatively, use commercially available solutions with the appropriate and required specification. The reagents described below are used to set the reagent concentrations defined in <u>9.2</u> in the reaction vessel.

ttps://standards.iteh.al/catalog/standards/sist/99a3f4e1-8b03-43fe-af6d-19dfd2c5df16/osist-pren-iso-18724-202 6.2 Water, with a specific electrical resistance \geq 18,2 M Ω cm (25 °C). The water shall not have a measurable content of chromium(VI) or interfering compounds above one third of the reporting limit (see ISO/TS 13530)

- of the method.
- 6.3 Sulfuric acid, $c(H_2SO_4) = 5 \text{ mol/l}$.
- 6.4 Ethanol, $c(C_2H_5OH) = 17,2 \text{ mmol/l}.$
- 6.5 Ammonium sulfate, (NH₄)₂SO₄.
- 6.6 Ammonium hydroxide solution, $c(NH_4OH) = 13,3 \text{ mol/l.}$

6.7 Ammonium hydroxide/ammonium sulfate solution.

Dissolve 3,3 g ammonium sulfate ($\underline{6.5}$) in approximately 75 ml of water ($\underline{6.2}$) in a 100 ml volumetric flask, add 6,5 ml ammonium hydroxide solution ($\underline{6.6}$) and dilute to volume with water ($\underline{6.2}$).

The solution contains 250 mmol/l ammonium sulfate and 865 mmol/l ammonium hydroxide.

The solution is stable for one month if stored at room temperature in polyethylene or glass bottles.