

SLOVENSKI STANDARD

SIST EN ISO 10304-3:1998

01-januar-1998

**Kakovost vode - Določevanje raztopljenih anionov z ionsko kromatografijo - 3. del:
Določevanje kromata, jodida, sulfita, tiocianita in tiosulfata (ISO 10304-3:1997)**

Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 3: Determination of chromate, iodide, sulfite, thiocyanate and thiosulfate (ISO 10304-3:1997)

Wasserbeschaffenheit - Bestimmung der gelösten Anionen mittels Ionenchromatographie - Teil 3: Bestimmung von Chromat, Iodid, Sulfit, Thiocyanat und Thiosulfat (ISO 10304-3:1997)

Qualité de l'eau - Dosage des anions dissous par chromatographie des ions en phase liquide - Partie 3: Dosage des ions chromate, iode, sulfite, thiocyanate et thiosulfate (ISO 10304-3:1997)

Ta slovenski standard je istoveten z: EN ISO 10304-3:1997

ICS:

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

EN ISO 10304-3

NORME EUROPÉENNE

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August 1997

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Descriptors: see ISO document

English version

**Water quality - Determination of dissolved anions
by liquid chromatography of ions - Part 3:
Determination of chromate, iodide, sulfite,
thiocyanate and thiosulfate (ISO 10304-3:1997)**

Qualité de l'eau - Dosage des anions dissous
par chromatographie des ions en phase liquide
- Partie 3: Dosage des ions chromate, iode,
sulfite, thiocyanate et thiosulfate
(ISO 10304-3:1997)

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This European Standard was approved by CEN on 1997-07-18. 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.

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CEN

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

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

Foreword

The text of the International Standard from Technical Committee ISO/TC 147 "Water quality" of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 230 "Water analysis", the secretariat of which is held by DIN.

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 month of February 1998, and conflicting national standards shall be withdrawn at the latest by February 1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 10304-3:1997 has been approved by CEN as a European Standard without any modification.

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EN ISO 10304-3:1997

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NOTE: Normative references to International Standards are listed in annex ZA (normative).

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Annex ZA (normative)**Normative references to international publications
with their relevant European publications**

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 amendments 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.

<u>Publication</u>	<u>Year</u>	<u>Title</u>	<u>EN</u>	<u>Year</u>
ISO 5667-1	1980	Water quality - Sampling - Part 1: Guidance on the design of sampling programmes	EN 25667-1	1993
ISO 5667-2	1991	Water quality - Sampling - Part 2: Guidance on sampling techniques	EN 25667-2	1993
ISO 5667-3	1994	Water quality - Sampling - Part 3: Guidance on the preservation and handling of samples	EN ISO 5667-3	1995
ISO 10304-1	1994	Water quality - Determination of dissolved fluoride, chloride, nitrite, orthophosphate, bromide, nitrate and sulfate ions, using liquid chromatography of ions - Part 1: Method for water with low contamination	EN ISO 10304-1	1995
ISO 10304-2	1995	Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 2: Determination of bromide, chloride, nitrate, nitrite, orthophosphate and sulfate in waste water	EN ISO 10304-2	1996

INTERNATIONAL STANDARD

ISO
10304-3

First edition
1997-08-15

Water quality — Determination of dissolved anions by liquid chromatography of ions —

Part 3:

Determination of chromate, iodide, sulfite,
thiocyanate and thiosulfate

iTeh STANDARD PREVIEW

*Qualité de l'eau — Dosage des anions dissous par chromatographie des ions
en phase liquide —*

Partie 3: Dosage des ions chromate, iodure, sulfite, thiocyanate et thiosulfate

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10304-3 was prepared by Technical Committee ISO/TC 147, *Water quality*, SC 2, *Physical, chemical and biochemical methods*.

ISO 10304 consists of the following parts, under the general title *Water quality — Determination of dissolved anions by liquid chromatography of ions*:

- *Part 1: Determination of fluoride, chloride, bromide, nitrate, nitrite, orthophosphate and sulfate in water with low contamination*
- *Part 2: Determination of bromide, chloride, nitrate, nitrite, orthophosphate and sulfate in waste water*
- *Part 3: Determination of chromate, iodide, sulfite, thiocyanate and thiosulfate*
- *Part 4: Determination of chlorate, chloride and chlorite in water with low contamination*

Annexes A and B of this part of ISO 10304 are for information only.

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Introduction

The essential minimum requirements of an ion chromatographic system applied within the scope of this part of ISO 10304 are the following:

- | | |
|---------------------------------|--|
| a) Resolution of the column: | For the anion to be determined it is essential that the peak resolution does not fall below $R = 1,3$ (4.2.2, figure 3) |
| b) Method of detection: | 1) measurement of the electrical conductivity with or without suppressor device

2) spectrometric measurement (UV/VIS), directly or indirectly

3) amperometric direct detection |
| c) Applicability of the method: | Working ranges according to table 1 |
| d) Calibration (4.5.1): | Calibration and determination of the linear working range (see ISO 8466-1) |

Guaranteeing the analytical quality:	Validity check of the calibration function. Replicate determinations if necessary.
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The diversity of the appropriate and suitable assemblies and the procedural steps depending on them permit a general description only.

For further information on the analytical technique, see reference [1].

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Water quality – Determination of dissolved anions by liquid chromatography of ions – Part 3: Determination of chromate, iodide, sulfite, thiocyanate and thiosulfate

1 Scope

This part of ISO 10304 specifies methods for the determination in aqueous solution of the dissolved anions

- iodide, thiocyanate and thiosulfate (clause 4);
- sulfite (clause 5);
- chromate (clause 6).

An appropriate pretreatment of the sample (e.g. dilution) and the application of a conductivity detector (CD), UV detector (UV) or amperometric detector (AD) make the working ranges given in table 1 feasible.

Table 1 — Applicable working ranges

Anion	Working range ¹⁾	Detector
Chromate (CrO ₄), clause 6	0,05 mg/l to 50 mg/l	UV (λ = 365 nm)
Iodide (I), clause 4	0,1 mg/l to 50 mg/l	CD or UV (λ = 205 nm to 236 nm) AD (approximately 0,7 V to 1,1 V)
Sulfite (SO ₃), clause 5	0,1 mg/l to 50 mg/l	CD
Thiocyanate (SCN), clause 4	0,5 mg/l to 50 mg/l 0,1 mg/l to 50 mg/l	UV (λ = 205 nm to 220 nm) CD or UV (λ = 205 nm to 220 nm) AD (approximately 0,7 V to 1,1 V)
Thiosulfate (S ₂ O ₃), clause 4	0,1 mg/l to 50 mg/l	CD or UV (λ = 205 nm to 220 nm) AD (approximately 0,7 V to 1,1 V)
¹⁾ The working range is restricted by the exchange capacity of the columns. Dilute the sample into the working range if necessary.		

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 10304. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5667-1:1980	<i>Water quality - Sampling - Part 1: Guidance on the design of sampling programmes.</i>
ISO 5667-2:1991	<i>Water quality - Sampling - Part 2: Guidance on sampling techniques.</i>
ISO 5667-3:1994	<i>Water quality - Sampling - Part 3: Guidance on the preservation and handling of samples.</i>
ISO 8466-1:1990	<i>Water quality - Calibration and evaluation of analytical methods and estimation of performance characteristics -Part 1: Statistical evaluation of the linear calibration function.</i>
ISO 10304-1:1992	<i>Water quality - Determination of dissolved fluoride, chloride, nitrite, orthophosphate, bromide, nitrate, and sulfate ions, using liquid chromatography of ions - Part 1: Method for water with low contamination.</i>
ISO 10304-2:1995	<i>Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 2: Determination of bromide, chloride, nitrate, nitrite, orthophosphate and sulfate in waste water.</i>

3 Principle

3.1 Separation of ions is carried out by liquid chromatography using a separating column. A low capacity anion exchanger is used as the stationary phase, and usually aqueous solutions of salts of weak monobasic and dibasic acids as mobile phases (eluent, see 4.1.16, 5.1.4, 6.1.9).

3.2 The addition of organic agents, such as 4-hydroxybenzonitrile (see 4.1.16.2.2, 4.3.4), or organic solvents to the eluent can be used to speed up the elution or reduce the tailing effects, especially for the analysis of the more strongly polarizable ions iodide, thiocyanate and thiosulfate.

3.3 Detection is by conductivity (CD), UV and amperometric detectors (AD).

3.3.1 When using conductivity detectors it is essential that the eluents have a sufficiently low conductivity. For this reason, conductivity detectors are often combined with suppressor devices (cation exchangers) which will reduce the conductivity of the eluents and transform the sample species into their respective acids.

3.3.2 UV detection measures either the absorption directly (see table 1) or, in the case of anions which are transparent in the UV-range, the decrease in the background absorption caused by a UV-absorbing eluent is measured (indirect measurement). If indirect UV-detection is used, the measuring wavelength depends on the composition of the eluent.

3.3.3 Amperometric detectors measure the quantity of current caused by the oxidation of anions. The oxidation voltage required for the anions in question depends on the pH value of the eluent.

3.4 The concentration of the respective anions is determined by a calibration of the overall procedure. Particular cases may require calibration by means of standard addition (spiking).

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4 Determination of iodide, thiocyanate and thiosulfate

Follow the instructions given in clause 4 to make the working ranges given in table 1 feasible.

4.1 Reagents

Use only reagents of recognized analytical grade, if commercially available. Carry out weighing with an accuracy of 1% of the nominal mass. The water shall have an electrical conductivity of < 0,01 mS/m and shall not contain particulate matter of a particle size > 0,45 µm. An increase of the electrical conductivity due to an uptake of carbon dioxide does not interfere with the determination.

4.1.1 Sodium hydrogen carbonate, NaHCO₃

4.1.2 Sodium carbonate, Na₂CO₃

4.1.3 Phthalic acid, C₈H₆O₄

4.1.4 Disodium tetraborate, Na₂B₄O₇

4.1.5 Gluconic acid, sodium salt, C₆H₁₁NaO₇

4.1.6 Methanol, CH₃OH

4.1.7 Boric acid, H₃BO₃

4.1.8 Glycerol, C₃H₈O₃