

SVENSK STANDARD

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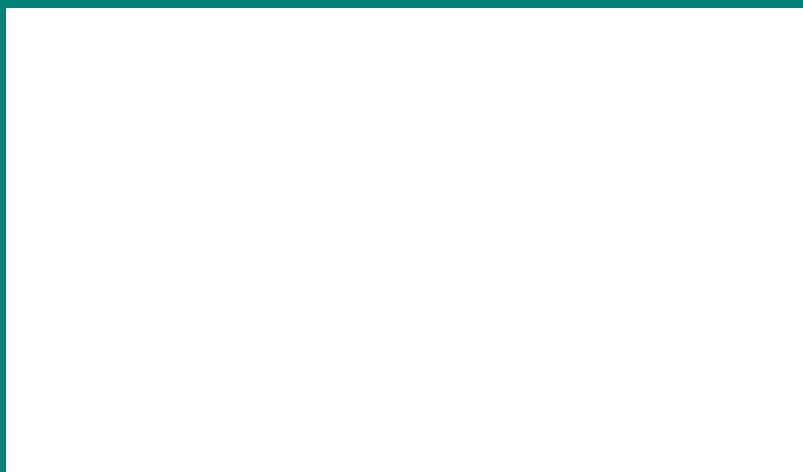
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Vattenundersökningar – Bestämning av lösta anjoner med jonkromatografi –

Del 1: Bestämning av bromid, klorid, fluorid, nitrat, nitrit, fosfat och sulfat (ISO 10304-1:2007)

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

Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007)



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Denna standard ersätter SS-EN ISO 10304-1, utgåva 1 och SS-EN ISO 10304-2, utgåva 1.

The European Standard EN ISO 10304-1:2009 has the status of a Swedish Standard. This document contains the official English version of EN ISO 10304-1:2009.

This standard supersedes the Swedish Standard SS-EN ISO 10304-1, edition 1 and SS-EN ISO 10304-2, edition 1.

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 10304-1

March 2009

ICS 13.060.50

Supersedes EN ISO 10304-1:1995, EN ISO 10304-2:1996

English Version

Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007)

Qualité de l'eau - Dosage des anions dissous par chromatographie des ions en phase liquide - Partie 1: Dosage du bromure, chlorure, fluorure, nitrate, nitrite, phosphate et sulfate (ISO 10304-1:2007)

Wasserbeschaffenheit - Bestimmung von gelösten Anionen mittels Flüssigkeits-Ionenchromatographie - Teil 1: Bestimmung von Bromid, Chlorid, Fluorid, Nitrat, Nitrit, Phosphat und Sulfat (ISO 10304-1:2007)

This European Standard was approved by CEN on 1 March 2009.

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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 Management Centre has the same status as the official versions.

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Foreword

The text of ISO 10304-1:2007 has been prepared by Technical Committee ISO/TC 147 "Water quality" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 10304-1:2009 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 September 2009, and conflicting national standards shall be withdrawn at the latest by September 2009.

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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, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of ISO 10304-1:2007 has been approved by CEN as a EN ISO 10304-1:2009 without any modification.

Introduction

The user should be aware that particular problems could require the specification of additional conditions not provided for in this part of ISO 10304.

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

Part 1:

Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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 and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this International Standard be carried out by suitably trained staff.

1 Scope

This part of ISO 10304 specifies a method for the determination of dissolved bromide, chloride, fluoride, nitrate, nitrite, orthophosphate and sulfate in water, e.g. drinking water, ground water, surface water, waste water, leachates and marine water by liquid chromatography of ions.

The lower limit of application is $\geq 0,05$ mg/l for bromide and for nitrite, and $\geq 0,1$ mg/l for chloride, fluoride, nitrate, orthophosphate, and sulfate. The lower limit of application depends on the matrix and the interferences encountered.

The working range may be expanded to lower concentrations (e.g. $\geq 0,01$ mg/l) if an appropriate pre-treatment of the sample (e.g. conditions for trace analyses, pre-concentration technique) is applied, and/or if an ultraviolet (UV) detector (for bromide, nitrate and nitrite) is used.

2 Normative references

The following referenced documents are indispensable for the application 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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of water samples*

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*

3 Interferences

3.1 Organic acids

Aliphatic organic acids such as mono- or dicarboxylic acids may interfere with the separation of the anions.

3.2 Sulfite

Sulfite may cause a positive bias for sulfate due to autooxidation. In this case the sample may be adjusted to pH 10 and formaldehyde solution is added in order to stabilize sulfite, if necessary.

3.3 Metals

The presence of metals (e.g. alkaline earth metals, transition metals, heavy metals) possibly interfering with the anions of interest, should be checked and can be eliminated with the aid of special cation exchangers (e.g. cartridge in the H-form or Na-form).

NOTE Depending on the sample matrix, the use of cation exchangers in the H-form can cause losses of fluoride and nitrite.

4 Principle

The sample is pretreated in order to remove solids (see Clause 7), sulfite and metal ions, if necessary. The anions of interest (bromide, chloride, fluoride, nitrate, nitrite, orthophosphate and sulfate), are separated by liquid chromatography, applying an anion exchange resin as stationary phase, and aqueous solutions of salts of weak mono- and dibasic acids as eluents for isocratic or gradient elution (e.g. carbonate, hydrogencarbonate, hydroxide eluent) (5.10). Detection is carried out using a conductivity detector (CD).

When using CDs it is essential that the eluents show a sufficiently low conductivity. For this reason, CDs are usually combined with a suppressor device (cation exchanger), which will reduce the conductivity of the eluent and transform the sample species into their respective acids.

NOTE A UV detector is not required to carry out this analysis, but can be used for bromide, nitrate or nitrite if a higher sensitivity is required and/or in case of a matrix interference to the CD. If used, bromide, nitrate and nitrite can be measured at $\lambda = 200$ nm to $\lambda = 215$ nm.

Check resolution, R , to ensure that it complies with the required separation conditions (6.2). UV may be used in combination with a CD. UV measures the absorption directly.

Calibration is carried out as specified in ISO 8466-1 or ISO 8466-2 (8.2). In special cases, extended working ranges (e.g. two concentration decades) may be applied.

Control experiments are necessary to check the validity of the calibration function. Replicate determinations may be necessary. Use of the method of standard addition may be required when matrix interferences are expected (8.3).

5 Reagents

Use only reagents of recognized analytical grade. Weigh the reagents with an accuracy of ± 1 % of the nominal mass, unless stated otherwise. The reagents listed in 5.2 to 5.5 may be considered representative examples for the preparation of eluents (5.10).

5.1 Water, complying with grade 1, as defined in ISO 3696.

5.2 Sodium hydrogencarbonate, NaHCO_3 .

5.3 Sodium carbonate, Na₂CO₃.

5.4 Sodium hydroxide, NaOH.

5.5 Potassium hydroxide, KOH.

5.6 Bromide, chloride, fluoride, nitrate, nitrite, orthophosphate and sulfate stock standard solutions, ρ = 1 000 mg/l each.

Single anion and mixed anion stock solutions with adequate and required specification are commercially available. These solutions are considered to be stable for several months.

For an alternative preparation of stock solutions see Table 1. Dissolve the salts accordingly, after appropriate treatment.

Table 1 — Mass portion and pre-treatment for stock solutions

Anion to be determined	Salt to be used ^a	Mass g	Pre-treatment by drying at (105 ± 5) °C for at least
Bromide	NaBr	1,287 7	6 h
Chloride	NaCl	1,648 4	2 h
Fluoride	NaF	2,210 0	1 h
Nitrate	NaNO ₃	1,370 7	24 h
Nitrite	NaNO ₂	1,499 8	1 h
Orthophosphate	KH ₂ PO ₄	1,433 0	1 h
Sulfate	Na ₂ SO ₄	1,478 6	1 h

^a Alternative salts with adequate and required specification may be used.

5.7 Bromide, chloride, fluoride, nitrate, nitrite, orthophosphate and sulfate standard solutions

5.7.1 General

Depending on the concentrations expected, prepare single or mixed standard solutions, of bromide, chloride, fluoride, nitrate, nitrite, orthophosphate and sulfate concentrations from the stock standard solution (5.6). Store the standard solutions in polyethene bottles.

5.7.2 Example for a bromide, chloride, fluoride, nitrate, nitrite, orthophosphate and sulfate mixed standard solution, ρ = 10 mg/l each.

Pipette 1,0 ml of each of the stock standard solutions (5.6) into a 100 ml volumetric flask, and dilute to volume with water (5.1).

The solutions are stable for 1 week, if stored in the dark at 2 °C to 8 °C in polyethene bottles.

5.8 Bromide, chloride, fluoride, nitrate, nitrite, orthophosphate and sulfate calibration solutions

Depending on the concentrations expected in the sample, use the standard solution (5.7.2) to prepare e.g. 5 to 10 calibration solutions distributed as evenly as possible over the expected working range.

For example, proceed as follows for the range 0,05 mg/l to 0,5 mg/l: