Vee kvaliteet. Lahustunud anioonide määramine ioonvahetus-vedelikkromatograafiat kasutades. Osa 2: Bromiidi, kloriidi, nitraadi, nitriti, ortofosfaadi ja sulfaadi määramine heitvees

Water quality - Determination of dissolved anions by liquid chromotagraphy of ions - Part 2: Determination of bromide, chloride, nitrate, nitrite, orthophosphate and sulfate in waste water

EESTI STANDARDI EESSÕNA **NATIONAL FOREWORD**

Käesolev Eesti standard EVS-EN ISO 10304-2:1999 sisaldab Euroopa standardi EN ISO 10304-2:1996 ingliskeelset teksti. Käesolev dokument on jõustatud 12.12.1999 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes. Standard on kättesaadav Eesti standardiorganisatsioonist.	This Estonian standard EVS-EN ISO 10304-2:1999 consists of the English text of the European standard EN ISO 10304-2:1996. This document is endorsed on 12.12.1999 with the notification being published in the official publication of the Estonian national standardisation organisation. The standard is available from Estonian standardisation organisation.

Käsitlusala: Standardi käesolev osa määrab kindlaks meetodi lahustunud anioonide - bromiidi, kloriidi, nitraadi, nitriti, ortofosfaadi ja sulfaadi - määramiseks heitvees.	**Scope:**

ICS 13.060.30, 13.060.50

Võtmesõnad: anioonid, bromiidid, keemiline analüüs, kloriidid, kvaliteet, lahustuv aine, nitraadid, nitritid, ortofosfaadid, reovesi, sisalduse määramine, vee saastumine, veetestid, vesi

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Descriptors: Water analysis, anions, waste water.

English version

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
(ISO 10304-2:1995)

Qualité de l'eau – Dosage des anions dissous par chromatographie des ions en phase liquide – Partie 2: Dosage des ions bromure, chlorure, nitrate, nitrite, orthophosphate et sulfate dans les eaux usées (ISO 10304-2:1995)

Wasserbeschaffenheit – Bestimmung der gelösten Anionen mittels Ionenchromatographie – Teil 2: Bestimmung von Bromid, Chlorid, Nitrat, Nitrit, Orthophosphat und Sulfat in Abwasser (ISO 10304-2:1995)

This European Standard was approved by CEN on 1996-07-05 and is identical to the ISO Standard as referred to.

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.

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 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 the United Kingdom.

CEN

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

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Foreword

International Standard

ISO 10304-2:1995 Water quality – Determination of dissolved anions by liquid chromatography of ions – Determination of bromide, chloride, nitrate, nitrite, orthophosphate and sulfate in waste water,

which was prepared by ISO/TC 147 'Water quality' of the International Organization for Standardization, has been adopted by Technical Committee CEN/TC 230 'Water analysis' as a European Standard.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, and conflicting national standards withdrawn, by February 1997 at the latest.

In accordance with the CEN/CENELEC Internal Regulations, 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, and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 10304-2:1995 was approved by CEN as a European Standard without any modification.

NOTE: Normative references to international publications are listed in Annex ZA (normative).

Introduction

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

Resolution power

It is essential that the peak resolution R does not fall below 1,3 for the anion to be determined and the nearest peak (see clause 6 and figure 3).

Method of detection

a) Measurement of the electrical conductivity with or without a suppressor device, respectively.

b) Photometric measurement (UV/VIS), directly or indirectly.

Applicability of the method

Working ranges are according to table 1.

Calibration

Calibration and determination of the linear working range. Use of the method of standard addition for special cases of application (see clause 9).

Quality control of the analytical method

Validity check of the calibration function (see 8.3). Replicate determinations if necessary.

The diversity of the appropriate and suitable assemblies and the procedural steps depending on them (e.g. composition of the mobile phases) permit a global description only.

For further information on the analytical technique, refer to ISO 10304-1.

1 Scope

1.1 General

This part of ISO 10304 specifies a method for the determination of the dissolved anions bromide, chloride, nitrate, nitrite, orthophosphate and sulfate in waste waters.

The working ranges listed in table 1 may be obtained by an appropriate sample pretreatment (e.g. dilution) and by applying a conductivity detector (CD) or a UV-detector.

Table 1 — Working ranges of the analytical method

Anion	Working range mg/l	Detection direct UV-detection
Bromide (Br^-)	0,05 to 20	CD or UV (200 nm to 215 nm)
Chloride (Cl^-)	0,1 to 50	CD
Nitrate (NO_3^-)	0,1 to 50	CD or UV (200 nm to 215 nm)
Nitrite (NO_2^-)	0,05 to 20	CD or UV (200 nm to 215 nm)
Orthophosphate (PO_4^{3-})	0,1 to 20	CD
Sulfate (SO_4^{2-})	0,1 to 100	CD
NOTE — The working range is limited by the exchange capacity of the columns.		

1.2 Interferences

1.2.1 Organic acids, such as monocarboxylic or dicarboxylic acids, can interfere with the determination of inorganic anions.

1.2.2 In a buffered eluent (e.g. carbonate/hydrogen carbonate), the determination will not be influenced by the sample pH in the range of pH 2 to pH 9.

1.2.3 Large concentration differences between the anions Br^-, Cl^-, NO_3^-, NO_2^-, PO_4^{3-} and SO_4^{2-} may lead to typical cross-sensitivity interferences caused by an insufficient separation. The respective concentrations given in table 2 were typical for conductivity detectors and UV-detectors; no interferences could be observed with a sample volume of 50 µl. The data given are valid only when the quality requirements specified for the columns are met (see clause 6). The determination of chloride may be subject to interference from high fluoride concentrations.

Table 2 — Cross-sensitivity of anions
[Detection: conductivity (CD) and direct UV]

Ratio of the mass concentrations solute/interfering ion		Maximum tolerable absolute concentration of interfering ions[1] mg/l	
Br^-/Cl^-	1:500	Cl^-	500
Br^-/PO_4^{3-}	1:100	PO_4^{3-}	100
Br^-/NO_3^-	1:50	NO_3^-	100
Br^-/SO_4^{2-}	1:500	SO_4^{2-}	500
Br^-/SO_3^{2-}	1:50[2]		
Cl^-/NO_2^-	1:50	NO_2^-	5
Cl^-/NO_3^-	1:500	NO_3^-	500
Cl^-/SO_4^{2-}	1:500	SO_4^{2-}	500
NO_3^-/Br^-	1:100	Br^-	100
NO_3^-/Cl^-	1:500 (CD)	Cl^-	500
	1:2 000 (UV)	Cl^-	500
NO_3^-/SO_4^{2-}	1:500 (CD)	SO_4^{2-}	500
	1:1 000 (UV)	SO_4^{2-}	500
NO_3^-/SO_3^{2-}	1:50[2]		
NO_2^-/Cl^-	1:250 (CD)	Cl^- (CD)	100
	1:10 000 (UV)	Cl^- (UV)	500
NO_2^-/PO_4^{3-}	1:50	PO_4^{3-}	20
NO_2^-/NO_3^-	1:500	NO_3^-	500
NO_2^-/SO_4^{2-}	1:500 (CD)	SO_4^{2-}	500
	1:1 000 (UV)	SO_4^{2-}	500
PO_4^{3-}/Br^-	1:100	Br^-	100
PO_4^{3-}/Cl^-	1:500	Cl^-	500
PO_4^{3-}/NO_3^-	1:500	NO_3^-	400
PO_4^{3-}/NO_2^-	1:100	NO_2^-	100
PO_4^{3-}/SO_4^{2-}	1:500	SO_4^{2-}	500
PO_4^{3-}/SO_3^{2-}	1:50[2]		
SO_4^{2-}/Cl^-	1:500	Cl^-	500
SO_4^{2-}/NO_3^-	1:500	NO_3^-	400
SO_4^{2-}/SO_3^{2-}	1:50[2]		
$SO_4^{2-}/S_2O_3^{2-}$	1:500		
SO_4^{2-}/I^-	1:500		

1) Dilute the sample if the interfering concentration is exceeded.

2) When it is present, SO_3^{2-} will always interfere.

1.2.4 The determination of sulfate may be subject to interference by high iodide or thiosulfate concentrations.

Relations: SO_4^{2-}/I^- or $SO_4^{2-}/S_2O_3^{2-}$ or $I^-/S_2O_3^{2-}$ = 1: > 500

Cross-sensitivities to other anions, such as Br^-, Cl^-, NO_3^-, NO_2^-, PO_4^{3-} and SO_4^{2-}, may occur, especially in the presence of sulfite ions whose retention strongly depends on the selectivity of the separating column used. Inorganic anions such as fluoroborate or chlorite can interfere with the determination of the named inorganic anions.

NOTE 1 The identification of some anions (e.g. nitrite) or the detection of interferences (e.g. fatty acids) can be facilitated by using a conductivity detector and UV-detector placed in series.

Anion combinations (e.g. Cl^-/I^-) which are not listed in table 2 will not interfere in the specified range of application.

Solid particles and organic compounds (such as mineral oils, detergents and humic acids) shorten the lifetime of the separating column and are therefore eliminated before the analysis is started (see clause 7).

Sulfide ions can cause errors during the determination of sulfate; they are eliminated according to clause 7.

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 part of ISO 10304 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, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes.*

ISO 5667-2:1991, *Water quality — Sampling — Part 2: Guidance on sampling techniques.*

ISO 5667-3:1994, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples.*