

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**

The European Standard EN ISO 10304-2:1996 has the status of a
British Standard

ICS 13.060.01

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- The Association of the Laboratory Supply Industry
- British Agrochemicals Association Ltd.
- British Ceramic Research
- British Gas plc
- British Soft Drinks Association Ltd.
- Chartered Institution of Water and Environmental Management
- Chemical Industries' Association
- Convention of Scottish Local Authorities
- Department of the Environment (Water Directorate)
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- Swimming Pool and Allied Trades Association Ltd.
- Water Companies Association
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- Water Services Association of England and Wales

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National foreword

This British Standard has been prepared by Subcommittee EH/3/2 and is the English language version of EN ISO 10304-2:1996 *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* published by the European Committee for Standardization (CEN), which endorses ISO 10304-2:1996, published by the International Organization for Standardization (ISO).

The tests described in this British Standard should only be carried out by suitably qualified persons with an appropriate level of chemical expertise. Standard chemical procedures should be followed throughout.

Cross-references

Publication referred to	Corresponding British Standard
EN 25667-1:1993 (ISO 5667-1:1980)	BS EN 25667/BS EN ISO 5667 <i>Water quality. Sampling</i> BS EN 25667-1:1994 <i>Guidance on the design of sampling programmes</i>
EN 25667-2:1993 (ISO 5667-2:1991)	BS EN 25667-2:1993 <i>Guidance on sampling techniques</i>
EN ISO 5667-3:1995 (ISO 5667-3:1994)	BS EN ISO 5667-3:1996 <i>Guidance on the preservation and handling of samples</i>
EN ISO 10304-1:1995 (ISO 10304-1:1992)	BS EN ISO 10304 <i>Water quality — Determination of dissolved fluoride, chloride, nitrite, orthophosphate, bromide, nitrate and sulfate ions, using liquid chromatography of ions</i> BS EN ISO 10304-1: 1995 <i>Method for water with low contamination</i>

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 18, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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Descriptors: Water, sewage, quality, water pollution, soluble matter, water tests, chemical analysis, determination of content, anions, bromides, chlorides, nitrates, nitrites, orthophosphates, sulfates, high performance liquid chromatography

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, dan les eaux usées
(ISO 10304-2:1995)

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

This European Standard was approved by CEN on 1996-07-05. 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.

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CEN

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

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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 February 1997, and conflicting national standards shall be withdrawn at the latest by February 1997.

According 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 and the United Kingdom.

NOTE Normative references to International Standards 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

- Measurement of the electrical conductivity with or without a suppressor device, respectively.
- 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 ^a 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 ^b	
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) 1 : 2 000 (UV)	Cl ⁻ 500 500
NO ₃ ⁻ /SO ₄ ²⁻ 1 : 500 (CD) 1 : 1 000 (UV)	SO ₄ ²⁻ 500 500
NO ₃ ⁻ /SO ₃ ²⁻ 1 : 50 ^b	
NO ₂ ⁻ /Cl ⁻ 1 : 250 (CD) 1 : 10 000 (UV)	Cl ⁻ (CD) 100 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) 1 : 1 000 (UV)	SO ₄ ²⁻ 500 500
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 ^b	
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 ^b	
SO ₄ ²⁻ /S ₂ O ₃ ²⁻ 1 : 500	
SO ₄ ²⁻ /I ⁻ 1 : 500	

^a Dilute the sample if the interfering concentration is exceeded.

^b When it is present, SO₃²⁻ will always interfere.

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

Relations: SO₄²⁻/I⁻ or SO₄²⁻/S₂O₃²⁻ or I⁻/S₂O₃²⁻
= 1 : > 500

Cross-sensitivities to other anions, such as Br⁻ Cl⁻ NO₃⁻, NO₂⁻, PO₄³⁻ and SO₄²⁻, 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*.

ISO 8466-1:1990, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*.

ISO 10304-1:1992, *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*.