

BS EN ISO 14403-2:2012



BSI Standards Publication

Water quality — Determination of total cyanide and free cyanide using flow analysis (FIA and CFA)

Part 2: Method using continuous flow analysis (CFA) (ISO 14403-2:2012)

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National foreword

This British Standard is the UK implementation of EN ISO 14403-2:2012. It supersedes BS EN ISO 14403:2002 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee EH/3/2, Physical chemical and biochemical methods.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

Water quality - Determination of total cyanide and free cyanide
using flow analysis (FIA and CFA) - Part 2: Method using
continuous flow analysis (CFA) (ISO 14403-2:2012)

Qualité de l'eau - Dosage des cyanures totaux et des
cyanures libres par analyse en flux continu (FIA et CFA) -
Partie 2: Méthode par analyse en flux continu (CFA) (ISO
14403-2:2012)

Wasserbeschaffenheit - Bestimmung von Gesamtcyanid
und freiem Cyanid mittels Fließanalytik (FIA und CFA) - Teil
2: Verfahren der kontinuierlichen Durchflussanalyse (CFA)
(ISO 14403-2:2012)

This European Standard was approved by CEN on 13 July 2012.

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN ISO 14403-2:2012) has been prepared by Technical Committee ISO/TC 147 "Water quality" in collaboration with 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of an identical text or by endorsement, at the latest by January 2013, and conflicting national standards shall be withdrawn at the latest by January 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN ISO 14403:2002.

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

Endorsement notice

The text of ISO 14403-2:2012 has been approved by CEN as a EN ISO 14403-2:2012 without any modification.

Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Interferences	2
4.1 Interferences by oxidizing agents	2
4.2 Interferences by sulfide, sulfite, nitrite and carbonyl compounds	2
4.3 Other interferences	2
5 Principle	3
5.1 Determination of total cyanide	3
5.2 Determination of free cyanide	3
6 Reagents	3
7 Apparatus	6
8 Sampling and sample preparation	8
9 Procedure	9
9.1 Flow system set-up	9
9.2 Reagent blank measurement	9
9.3 Checking the suitability of the flow system	10
9.4 Calibration	10
9.5 Sample measurement	11
10 Calculation	11
11 Expression of results	11
12 Test report	11
Annex A (informative) Examples of flow systems	13
Annex B (normative) Determination of the real cyanide concentration in the potassium cyanide solution	16
Annex C (informative) Example for the determination of total cyanide and free cyanide by continuous flow analysis (CFA) with gas diffusion and amperometric detection	17
Annex D (informative) Performance data	19
Bibliography	21

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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ISO 14403-2 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

This first edition of ISO 14403-2 cancels and replaces ISO 14403:2002, which has been technically revised.

ISO 14403 consists of the following parts, under the general title *Water quality — Determination of total cyanide and free cyanide using flow analysis (FIA and CFA)*:

- *Part 1: Method using flow injection analysis (FIA)*
- *Part 2: Method using continuous flow analysis (CFA)*

Introduction

Methods using flow analysis automate wet chemical procedures and are particularly suitable for the processing of many analytes in water in large series of samples at a high frequency of analysis.

Analysis can be performed by flow injection analysis (FIA) or continuous flow analysis (CFA). Both methods share the feature of an automatic introduction of the sample into a flow system (manifold) in which the analytes in the sample react with reagent solutions on their way through the manifold. Sample preparation may be integrated in the manifold. The reaction product is measured in a flow detector (e.g. flow photometer).

See the foreword for a list of parts of this International Standard.

It should be investigated whether and to what extent particular problems require the specification of additional marginal conditions.

Water quality — Determination of total cyanide and free cyanide using flow analysis (FIA and CFA) —

Part 2: Method using continuous flow analysis (CFA)

WARNING — Persons using this part of ISO 14403 should be familiar with normal laboratory practice. This part of ISO 14403 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 part of ISO 14403 be carried out by suitably trained staff.

1 Scope

This part of ISO 14403 specifies methods for the determination of cyanide in various types of water (such as ground, drinking, surface, leachate, and waste water) with cyanide concentrations usually from 2 µg/l to 500 µg/l expressed as cyanide ions in the undiluted sample. The range of application can be changed by varying the operation conditions, e.g. by diluting the original sample or changing the pathlength of the flow cell.

In this method, a suitable mass concentration range from 10 µg/l to 100 µg/l is described.

Seawater can be analysed with possible changes in sensitivity and adaptation of the reagent and calibration solutions to the salinity of the samples.

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 and laboratory use — Specification and test methods*

ISO 5667-3, *Water quality — Sampling — Part 3: 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 Terms and definitions

For the purpose of this part of ISO 14403, the following definitions apply:

3.1

free cyanide

easily liberatable cyanide

sum of cyanide ions and the cyanide bound in weak metal cyanide complexes that liberate HCN at pH 3,8

3.2
total cyanide
free cyanide (3.1), and in addition stronger metal–cyanide complex compounds, with the exception of cyanide bound in gold, cobalt, platinum, ruthenium, and rhodium complexes, from which recovery can be partial

NOTE 1 Thiocyanate, organically bound cyanide and cyanate are not included in this definition of total cyanide.

NOTE 2 Distillation methods may recover some organic cyanide. Use the diffusion method when organic cyanide complexes are suspected to be present.

4 Interferences

4.1 Interferences by oxidizing agents

Oxidizing agents such as chlorine decompose most of the cyanides. If the presence of oxidizing agents cannot be excluded, treat the sample immediately after sampling. Test a drop of the sample with potassium iodide-starch test paper (KI starch paper); a blue colour indicates the need for treatment. Add sodium thiosulfate, a few crystals at a time, until a drop of sample produces no colour on the indicator paper. Then add an additional portion of 0,6 g of ascorbic acid (6.9) for each 1 000 ml of sample volume.

Do not add ascorbic acid unless samples will be analysed within 24 h.

4.2 Interferences by sulfide, sulfite, nitrite and carbonyl compounds

Interferences by sulfide start at 100 mg/l. It affects the colorimetric procedure, especially the gas diffusion method, and the amperometric procedure (see Annex C). If a drop of the sample on lead acetate test paper indicates the presence of sulfide, treat an additional 25 ml of the stabilized sample (pH >12) to that required for the cyanide determination with powdered lead carbonate.

Lead sulfide precipitates if the sample contains sulfide.

Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper.

Filter the solution through a dry filter paper into a dry beaker, and from the filtrate measure the sample to be used for analysis. Avoid a large excess of lead and a long contact time in order to minimize loss by complexation or occlusion of cyanide on the precipitated material.

If the amperometric method (Annex C) is applied, it is necessary systematically to add lead carbonate to samples during the analysis (a few milligrams for a 10 ml sample), followed by filtration or decantation performed before the filtered sample aliquot is placed on the sample tray of the continuous flow analyser.

Aldehydes and ketones can, under certain conditions, absorb cyanide by nucleophilic addition. To avoid this interference ethylenediamine can be added to the sample.

Under the given distillation conditions, aldehydes can transform cyanide to nitrite. Aldehydes can be removed by adding silver nitrate to the sample. The addition of AgNO₃ can alter the ratio of the concentrations of free and total cyanide. The user should evaluate this procedure.

Interference by nitrite above concentrations of 5 mg/l can be avoided by addition of sulfamic acid (6.10) to the buffer (pH 3,8) for the distillation and gas diffusion method (6.21.1).

Sulfite interferes above concentrations of 1 mg/l.

4.3 Other interferences

Samples containing particulate matter can lead to losses if the particles clog the transport tubes and are not transported completely into the UV unit. This effect can be minimized by homogenizing (e.g. stirring) the sample immediately prior to analysis to ensure that a representative sample is taken and to reduce the particle size. Remaining particles with diameters >0,1 mm should be removed by filtration.