# SVENSK STANDARD
# SS-EN ISO 14403-1:2012

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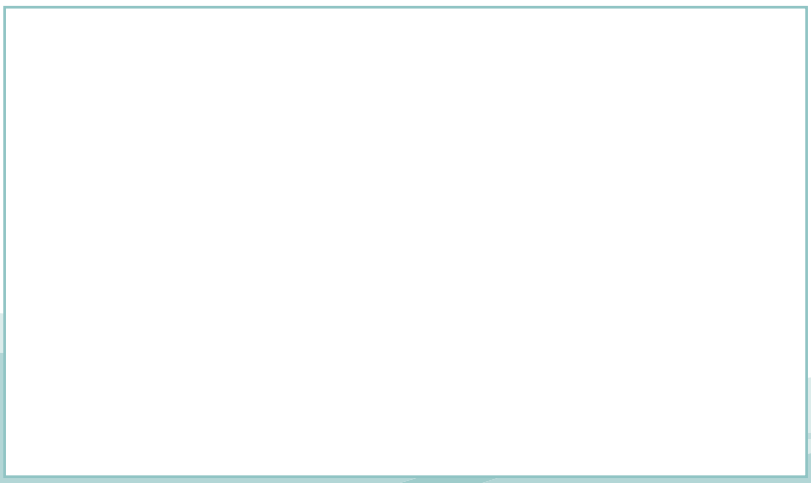
SWEDISH STANDARDS INSTITUTE

**Vattenundersökningar – Bestämning av totalhalt cyanid och fri cyanid genom flödesanalys (FIA och CFA) –**
**Del 1: Metod med flödesinjektion (FIA) (ISO 14403-1:2012)**

**Water quality – Determination of total cyanide and free cyanide using flow analysis (FIA and CFA) –**
**Part 1: Method using flow injection analysis (FIA)**
**(ISO 14403-1:2012)**



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Europastandarden EN ISO 14403-1:2012 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN ISO 14403-1:2012.

The European Standard EN ISO 14403-1:2012 has the status of a Swedish Standard. This document contains the official version of EN ISO 14403-1:2012.

**Förhållandet till övriga delar under samma huvudtitel - Utdrag ur Förord i ISO 14403-1:2012/ Relations to other parts under the same general title - Extract from the Foreword of ISO 14403-1:2012**

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)*



*Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.*

*Information about the content of the standard is available from the Swedish Standards Institute (SIS), telephone +46 8 555 520 00. Standards may be ordered from SIS Förlag AB, who can also provide general information about Swedish and foreign standards.*

Denna standard är framtagen av kommittén för Kemiska vattenundersökningar, SIS/TK 424.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på www.sis.se - där hittar du mer information.

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

# EN ISO 14403-1

July 2012

ICS 13.060.50

English Version

# Water quality - Determination of total cyanide and free cyanide using flow analysis (FIA and CFA) - Part 1: Method using flow injection analysis (FIA) (ISO 14403-1:2012)

Qualité de l'eau - Dosage des cyanures totaux et des cyanures libres par analyse en flux continu (FIA et CFA) - Partie 1: Méthode par analyse avec injection de flux (FIA) (ISO 14403-1:2012)

Wasserbeschaffenheit - Bestimmung von Gesamtcyanid und freiem Cyanid mittels Fließanalytik (FIA und CFA) - Teil 1: Verfahren mittels Fließinjektionsanalyse (FIA) (ISO 14403-1:2012)

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

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 CEN-CENELEC Management Centre 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 CEN-CENELEC Management Centre has the same status as the official versions.

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Ref. No. EN ISO 14403-1:2012: E

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# Foreword

This document (EN ISO 14403-1:2012) 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.

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 14403-1:2012 has been approved by CEN as a EN ISO 14403-1:2012 without any modification.

# 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 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 1:
## Method using flow injection analysis (FIA)

**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 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 using a different injection volume.

In this part of ISO 14403, a suitable mass concentration range from 20 µg/l to 200 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 purposes of this document, the following terms and 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, platinum, cobalt, ruthenium, and rhodium complexes from which recovery can be partial

# 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.

Carry out a holding time study at the sampling point in order to determine whether the sample is stable for the time period for preservation and whether the preservation is effective. If this preservation is ineffective, online measurement instrumentation may be required.

## 4.2 Interferences by sulfide, sulfite, nitrite, and carbonyl compounds

Interferences by sulfide start at 20 mg/l. 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.

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

Interference by nitrite occurs above concentrations of 2 mg/l and can be avoided by addition of sulfamic acid (6.8) to the buffer (pH 3,8) for the gas diffusion method (6.20.1).

Sulfite interferes above concentrations of 1 mg/l.

## 4.3 Other interferences

Particulate matter in the sample can lead to clogging of the transport tubes and interferes with the photometric measurement. Particles of diameter >0,1 mm should be removed by filtration.

Thiocyanate can slightly interfere and lead to positive bias (9.3.2). Significant interferences can arise from cyanide impurities in thiocyanate (6.16).

# 5 Principle

## 5.1 Determination of total cyanide

Complex-bound cyanide is decomposed by UV light at pH 3,8. A UV-B lamp (emission maximum >310 nm to 400 nm) and a digestion coil of perfluoro (ethylene/propylene) (FEP) or polytetrafluorethylene (PTFE) is used to filter off UV light with a wavelength <290 nm thus preventing the conversion of thiocyanate into cyanide. A hydrolytic treatment in a thermoreactor (85 °C) assists the decomposition.

The hydrogen cyanide present at pH 3,8 is separated by diffusion at 30 °C to 40 °C across a hydrophobic membrane. Hydrogen cyanide is absorbed in a sodium hydroxide solution.

The absorbed cyanide is then determined by the reaction of cyanide with chloramine-T to cyanogen chloride. This reacts with pyridine-4-carboxylic acid and 1,3-dimethylbarbituric acid to give a red dye.

### 5.2 Determination of free cyanide

During the procedure specified in 5.1, the UV-B lamp is switched off when determining the free cyanide content. A thermal decomposition with a citrate and succinate buffer is performed.

To liberate cyanide from the nickel complex, 50 µl tetraethylenepentamine solution (6.11) per 30 ml sample shall be added prior to the analysis (see Reference [8]).

For detection, see 5.1.

## 6 Reagents

**WARNING — KCN, $K_2Zn(CN)_4$, their solutions, and wastes are toxic. Waste containing these substances shall be removed appropriately.**

Use only reagents of recognized analytical grade.

Smaller portions of the following solutions can be applied provided the ratios of the prescribed volumes and mass concentrations are maintained.

**6.1 Water**, grade 1, as defined in ISO 3696.

**6.2 Hydrochloric acid**, $c$(HCl) = 1 mol/l.

**6.3 Sodium hydroxide solution I**, carrier solution, $c$(NaOH) = 0,4 mol/l (C2 in Figure A.1).

**6.4 Sodium hydroxide solution II**, $c$(NaOH) = 1,0 mol/l.

**6.5 Sodium hydroxide solution III**, $c$(NaOH) = 0,01 mol/l.

**6.6 Citric acid monohydrate**, $C_6H_8O_7 \cdot H_2O$.

**6.7 Succinic acid**, $C_4H_6O_4$.

**6.8 Sulfamic acid**, $H_3SO_3N$.

**6.9 Disodium ethylenediamine tetraacetic acid**, $Na_2EDTA$, $C_{10}H_{14}N_2O_8Na_2$.

**6.10 Tetraethylenepentamine**, $C_8H_{23}N_5$.

**6.11 Tetraethylenepentamine solution** (for free cyanide only).

Dissolve 0,75 g of tetraethylenepentamine (6.10) in 250 ml of water.

This solution is stable for 1 month if stored at room temperature.

**6.12 Potassium cyanide**, KCN.

**6.13 Chloramine-T trihydrate**, $C_7H_7ClNNaO_2S \cdot 3H_2O$.

**6.14 1,3-Dimethylbarbituric acid**, $C_6H_8N_2O_3$.

**6.15 Pyridine-4-carboxylic acid**, $C_6H_5NO_2$.

**6.16 Potassium thiocyanate**, KSCN.