INTERNATIONAL STANDARD

ISO 14403-1

First edition
2012-07-15

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

Part 1:
Method using flow injection analysis (FIA)

Qualité de l'eau — Dosage des cyanures totaux et des cyanures libres par analyse en flux (FIA et CFA) —

Partie 1: Méthode par analyse avec injection de flux (FIA)

Reference number
ISO 14403-1:2012(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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.

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

ISO 14403-1 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

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 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**, $c(\text{HCl}) = 1$ mol/l.

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

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

6.5 **Sodium hydroxide solution III**, $c(\text{NaOH}) = 0,01$ 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.

6.17 **Potassium hexacyanoferrate(III)**, $K_3Fe(CN)_6$.

6.18 **Cyanide standards**.

6.18.1 **Potassium cyanide solution**, KCN, ρ_{CN} = 1 000 mg/l (see Annex B).

Dissolve 2 500 mg ± 1 mg of potassium cyanide, KCN (6.12), in sodium hydroxide solution III (6.5) in a 1 000 ml volumetric flask, and make up to volume with sodium hydroxide solution III (6.5).

This solution is stable for 6 months at 2 °C to 8 °C.

Alternatively, a potassium tetracyanozincate solution (6.18.2) may be used.

6.18.2 **Potassium tetracyanozincate solution**, $K_2Zn(CN)_4$, ρ_{CN} = 1 000 mg/l ± 2 mg/l, commercially available.

This solution is stable for 6 months at 2 °C to 8 °C.

6.18.3 **Cyanide solution I,** ρ_{CN} = 10 mg/l.

Pipette 1 ml of the potassium tetracyanozincate solution I (6.18.2) or 1 ml of the potassium cyanide solution (6.18.1) into a 100 ml volumetric flask, and make up to volume with sodium hydroxide solution III (6.5).

This solution is stable for 1 week if stored at 2 °C to 8 °C.

6.19 **Calibration solutions**.

Prepare at least five calibration solutions with cyanide concentrations, regularly distributed over the working range, by appropriate dilution of the cyanide solution I (6.18.3). If, for example, six calibration solutions should be prepared, proceed as follows.

Pipette 10 ml of the cyanide solution I (6.18.3) into a 100 ml volumetric flask, and make up to volume with sodium hydroxide solution III (6.5). This solution contains 1 mg/l cyanide.

Pipette, into 50 ml volumetric flasks, 1 ml, 3 ml, 5 ml, 6 ml, 8 ml, or 10 ml, respectively, of the previously mentioned 1 mg/l cyanide solution and make up to volume with sodium hydroxide solution III (6.5).

These solutions contain 20 µg/l, 60 µg/l, 100 µg/l, 120 µg/l, 160 µg/l, and 200 µg/l of cyanide, respectively [except for corrections in the concentration found on titration of the potassium cyanide solution (6.18.1), (see Annex B].

These solutions are stable for 2 d if stored in a refrigerator at 2 °C to 8 °C.

6.20 **Reagents for the determination of cyanide**.

6.20.1 **Buffer**, pH 3,8, for gas diffusion method.

Dissolve, in about 350 ml water (6.1), 10,5 g of sodium hydroxide, 12,0 g of Na_2EDTA (6.9), 15,2 g of succinic acid (6.7), 27,0 g of citric acid monohydrate (6.6), and 12,5 g of sulfamic acid (6.8). Dilute to 500 ml with water.

This solution is stable for 1 week if stored in a refrigerator (1 °C to 5 °C).

NOTE The decomposition is performed with a citrate and succinate buffer, because this buffer has a higher capacity at pH 3,8 than a pure citrate buffer. Citrate and EDTA are added, because of their ability to support the decomposition of hexacyanoferrate. EDTA also avoids the precipitation of insoluble cyanides in the thermoreactor. Sulfamic acid is added to remove nitrite (see 4.2).

6.20.2 **Buffer solution for the final photometric determination** (R1 in Figure A.1).

Dissolve 7,0 g of sodium hydroxide (NaOH) in 250 ml of water. Add 35,4 g of succinic acid (6.7) and dilute to 500 ml with water.

The solution has a pH of approximately 4,3. When mixed with sodium hydroxide solution I (6.3; C2 in Figure A.1) a pH of 5,2 shall be achieved.

This solution is stable for 1 week if stored in a refrigerator at 1 °C to 5 °C.

6.20.3 Chloramine-T trihydrate solution (R2 in Figure A.1).

Dissolve 0,14 g of chloramine-T (6.13) in 100 ml of water.

This solution is stable for 1 week if stored in a refrigerator at 1 °C to 5 °C.

For best results, prepare the solution daily.

6.20.4 Colour reagent (R3 in Figure A.1).

Carefully dissolve, in a 1 000 ml volumetric flask, 7,0 g of sodium hydroxide, NaOH, in about 500 ml of water (6.1). Add 16,8 g ± 0,1 g of 1,3-dimethylbarbituric acid (6.14), and 13,6 g ± 0,1 g of pyridine-4-carboxylic acid (6.15), and dilute to approximately 975 ml with water (6.1).

If necessary, adjust the solution to pH 5,2 with hydrochloric acid (6.2) or sodium hydroxide solution II (6.4).

Make up to 1 000 ml with water (6.1). Mix this solution intensively (e.g. by using a magnetic stirrer) for 1 h at 30 °C and then filter over a pleated filter (e.g. hardened ashless paper).

This solution is stable for 1 month if stored in a refrigerator at 2 °C to 5 °C.

6.21 Thiocyanate solution, calculated cyanide concentration: ρ_{CN} = 100 mg/l.

Dissolve, in a 1 000 ml volumetric flask, 373 mg ± 1 mg of potassium thiocyanate (6.16) in sodium hydroxide solution III (6.5), and make up to volume with sodium hydroxide solution III (6.5).

This solution is stable for 2 months if stored in an amber bottle and refrigerated at 1 °C to 5 °C.

6.22 Potassium hexacyanoferrate(III) solution, calculated cyanide concentration ρ_{CN} = 10 mg/l.

6.22.1 Concentration, ρ_{CN}: 1 000 mg/l.

Dissolve, in a 100 ml volumetric flask, 211 mg ± 1 mg of potassium hexacyanoferrate(III) (6.17) in sodium hydroxide solution III (6.5), and make up to volume with sodium hydroxide solution III (6.5).

6.22.2 Concentration, ρ_{CN}: 10 mg/l.

Make up to volume, in a 100 ml volumetric flask, 1 ml of 1 000 mg/l CN solution (6.22.1) with sodium hydroxide solution III (6.5).

This solution is stable for 2 months if stored in a refrigerator at 1 °C to 5 °C.

7 Apparatus

Usual laboratory apparatus and in particular the following.

7.1 Flow injection analysis system for gas diffusion method.

7.1.1 General. A suitable example of the system contains the components specified in 7.1.2 to 7.1.11 (see Figure A.1). Alternative systems are also applicable if the requirements of Clause 9 are achieved.

7.1.2 Autosampler, or other device allowing a reproducible introduction of the sample.

7.1.3 Reagent reservoirs.

7.1.4 Low pulsation pump, with specific chemically inert pump tubes, for flow rates as shown in Figure A.1 as an example.

7.1.5 UV-lamp, with:

- an emission maximum >310 nm to 400 nm;
- a power of 8 W to12 W;
- a digestion coil of FEP or PTFE, internal diameter 0,8 mm, length 5 000 mm, tube wall thickness at maximum 1 mm (e.g. 351 nm UV lamp with PTFE coil).

The equipment shall be such that no UV light with a wavelength below 290 nm can reach the sample flow to avoid decomposition of thiocyanate to cyanide.

7.1.6 Thermoreactor 1: digestion coil of FEP with 0,8 mm × 3 600 mm, tube wall thickness at maximum 1,5 mm with a temperature of 85 °C for total cyanide and 40 °C for free cyanide (see Figure A.1).

7.1.7 Gas diffusion cell, with hydrophobic semipermeable membrane made from polypropylene or PTFE, for example, typical thickness 90 µm to 200 µm, pore size 0,1 µm to 1 µm.

7.1.8 Manifold with highly reproducible dosing of sample and reagents, with appropriate transport systems and connection assemblies of chemically inert polymer.

7.1.9 Photometric detector, with flow cell, wavelength 600 nm ± 10 nm. An appropriate optical pathlength should be used to achieve a minimum absorbance (absolute value) of 0,005 for a 20 µg/l cyanide solution.

7.1.10 Recording unit (e.g. strip chart recorder, integrator, printer and plotter or a computer data system). In general, peak height signals are measured.

7.1.11 Thermoreactor 2, 65 °C, length 3 000 mm, internal diameter 0,8 mm (see Figure A.1).

7.2 Additional apparatus.

7.2.1 Lead acetate test paper, commercially available.

7.2.2 Membrane filter assembly, with membrane filters having a pore size of 0,45 µm.

7.2.3 pH measuring device.

8 Sampling and sample preparation

Immediately after sampling, adjust the pH of the water samples to 12 with sodium hydroxide solutions I to III (6.3 to 6.5) such that the quantity of added alkali yields a negligible dilution of the sample.

If necessary, remove particles of diameter >0,1 mm by filtration or decantation at the laboratory.

Test for interferences and treat if necessary (see Clause 4).

Analyse the sample in accordance with Clause 9 as soon as possible after sampling, but as specified in ISO 5667-3 at the latest within 7 d. Store the sample in the dark.

9 Procedure

9.1 Flow system set-up

Set up and adjust the flow analysis system according to Table 1 depending on the determination to be performed.

Table 1 — Adjustment of flow injection analysis system for gas diffusion methods

Procedure	Component or parameter	Setting for determination to be performed	
		Total cyanide	Free cyanide
UV decomposition	UV lamp (7.1.5)	on	off
	Thermal decomposition (in addition to the UV decomposition)	85 °C	40 °C
Gas diffusion	**Buffer**, pH 3,8, for gas diffusion method (6.20.1)	yes	yes
	Temperature	—	—
Final photometric determination	Colour reagent (6.20.4)	yes	yes
	pH	5,2	5,2
	Wavelength	590 nm to 610 nm	590 nm to 610 nm

9.2 Reagent blank measurement

Set the analysis system in operation by first pumping water through the system.

Wait for stabilization of the baseline and zero the baseline.

Run the reagents buffer (pH 3,8) for gas diffusion (6.20.1), carrier solution (6.3), buffer solution for the final photometric determination (6.20.2), chloramine-T solution (6.20.3), and colour reagent (6.20.4), respectively, through the system and measure the increase in absorbance against water.

If the absorbance per centimetre of cell pathlength changes by more than 0,003 cm^{-1}, it is possible that either the water or the reagent solutions are contaminated. Take appropriate measures to eliminate the interference.

NOTE If the photometric detector does not give absorbance readings, the absorbance can be determined with an external absorbance-measuring spectrometer.

9.3 Checking the suitability of the flow system

9.3.1 Minimum absorbance

Run a cyanide calibration solution (6.19) with a concentration of 100 µg/l.

This calibration solution shall produce an absorbance per centimetre of cell pathlength of at least 0,027 cm^{-1}.

See Note to 9.2.

9.3.2 Recovery rates

Prepare standard solutions of hexacyanoferrate(III), and thiocyanate respectively, by placing 2 ml of potassium hexacyanoferrate(III) solution (6.22), and 2 ml of thiocyanate solution (6.21) respectively, in 100 ml measuring flasks. Make each of the solutions up to volume with sodium hydroxide solution III (6.5).

These prepared solutions should contain:

Solution A	hexacyanoferrate(III):	200 µg/l CN, chemically bound
Solution B	thiocyanate:	2 000 µg/l CN, chemically bound

Measure the cyanide (CN) content of solutions A and B with the flow system adjusted to the free and total cyanide method.

Consider the system to be suitable for the measurement of total cyanide if the determined cyanide content of solution A is ≥90 % of the theoretical cyanide content. Lower yields (<90 %) indicate inadequate effectiveness of the UV decomposition.

For the determination of free cyanide, the previously mentioned recovery rate shall be ≤5 %.

For both determinations, the determined cyanide content of solution B shall be ≤1 % of the theoretical cyanide content.

9.4 Calibration

Select the working mode of the flow system and calibrate by sequentially applying the calibration solutions (6.19) and the blank solution.

Prior to the calibration, zero the instrument, following the manufacturer's instructions as long as they are in accordance with the specifications of this part of ISO 14403.

Determine the measured values from the calibration solutions.

The test conditions for the calibration and the measurement of samples (9.5) are the same. The magnitude of the measuring signal is proportional to the mass concentration of cyanide. Establish the regression line for the measuring series obtained.

Calibrate the flow system as specified in ISO 8466-1. In general, Equation (1) is appropriate (ISO 8466-1). If the linearity test described in ISO 8466-1 shows that the calibration curve is not linear, calculate the calibration curve as specified in ISO 8466-2.

The measured value for the calibration solutions, y, in terms of instrument related units (e.g. peak heights in centimetres or counts), is given by:

$$y = b\rho + a \qquad (1)$$

where

b is the slope of the calibration function, expressed in instrument-related units or micrograms per litre;

ρ is the mass concentration of the standard solutions, expressed in micrograms per litre;

a is the ordinate intercept, expressed in instrument-related units.

9.5 Sample measurement

Analyse the samples, pretreated according to Clause 8, in the same way as the calibration solutions with the flow injection system.

If the mass concentrations of the samples exceed the validity of the calibrated working range, dilute the samples.

Check the validity of the calibration function after each sample series, but at least after the measurement of 10 to 20 samples, using one calibration solution each for the lower and upper part of the working range.

Make a new calibration, if necessary.

10 Calculation

Determine the mass concentrations of the samples using the measured values, obtained as specified in 9.4 for the calibration solutions.

Calculate ρ using Equation (2):

$$\rho = \frac{y - a}{b} \tag{2}$$

For an explanation of the symbols, see 9.4.

11 Expression of results

Report the results to two significant figures at most.

EXAMPLE 1	ρ(total CN)	$2{,}9 \times 10^2$ µg/l
EXAMPLE 2	ρ(free CN)	45 µg/l

12 Test report

This test report shall contain at least the following information:

a) the test method used, together with a reference to this part of ISO 14403 (ISO 14403-1:2012);

b) all information necessary for identification of the sample;

c) the type of sample pretreatment;

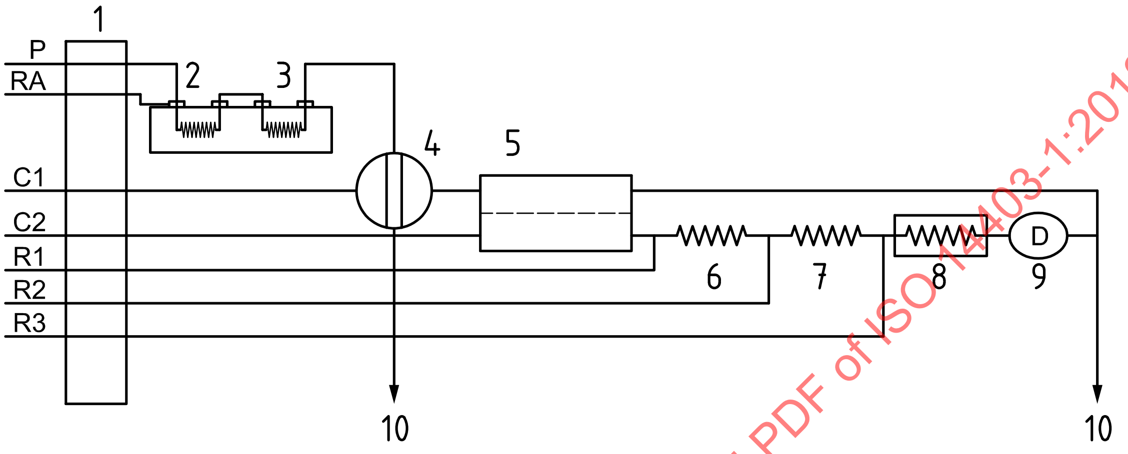
d) the cyanide concentration (free or total cyanide) in micrograms per litre, expressed in accordance with Clause 11;

e) any special observations noted during the determination;

f) any deviations from this part of ISO 14403, which could have affected the result.

Annex A
(informative)

Examples of flow systems



Key

1	pump (flow rates in ml/min)
2	UV lamp (7.1.5). For the determination of free cyanide the UV lamp is switched off.
3	thermoreactor 1, length 3 600 mm, internal diameter 0,8 mm, temperature 85 °C for total cyanide and 40 °C for free cyanide (7.1.6)
4	injector, injection volume: 400 µl
5	gas diffusion cell (7.1.7)
6	reaction coil, length 500 mm, internal diameter 0,5 mm
7	reaction coil, length 500 mm, internal diameter 0,8 mm
8	thermoreactor 2, 65 °C, length 3 000 mm, internal diameter 0,8 mm (7.1.11)
9	detector (e.g. 1 cm optical pathlength), wavelength 590 nm to 610 nm (7.1.9)
10	waste
P	sample:
	— for free cyanide: 0,51 ml/min;
	— for total cyanide: 0,32 ml/min.
RA	buffer (pH = 3,8) for gas diffusion method (6.20.1)
	— for free cyanide: 0,33 ml/min;
	— for total cyanide: 0,21 ml/min.
C1	200 ml buffer (pH = 3,8) for gas diffusion method (6.20.1) diluted to 500 ml with water: 1,08 ml/min
C2	carrier solution, sodium hydroxide solution I (6.3): 1,08 ml/min
R1	buffer solution for the final photometric determination (6.20.2): 0,75 ml/min
R2	chloramine-T trihydrate solution (6.20.3): 0,49 ml/min
R3	colour reagent (6.20.4): 1,08 ml/min

Figure A.1 — FIA system for the photometric determination of free and total cyanide (20 µg/l to 200 µg/l) with gas-diffusion separation (according to 7.1)

Annex B
(normative)

Determination of the real cyanide concentration in the potassium cyanide solution (6.18.1)

B.1 General

If KCN is used to prepare the cyanide calibration solutions (6.19), proceed as follows.

B.2 Additional reagents

B.2.1 *p*-Dimethylaminobenzylidene rhodanine.

B.2.2 Indicator solution.

Dissolve 0,02 g of *p*-dimethylaminobenzylidene rhodanine (B.2.1) in 100 ml of acetone (C_3H_6O).

This solution is stable for 1 week if stored in a refrigerator at 1 °C to 5 °C.

B.2.3 Silver nitrate solution, $c(\mathrm{AgNO_3})$ = 1 mmol/l.

B.3 Determination of cyanide concentration in potassium cyanide solution (6.18.1)

Pipette 10 ml of potassium cyanide solution (6.18.1) into a beaker. Add 0,25 ml of indicator solution (B.2.2).

Titrate with the silver nitrate solution (B.2.3) until the colour changes from yellow to yellow-red (consumption V_1).

Calculate the cyanide concentration in the potassium cyanide solution (6.18.1) using Equation (B.1):

$$\rho_{\mathrm{CN}} = \frac{V_1 c\left(\mathrm{AgNO_3}\right) M_{\mathrm{2CN}}}{V} \tag{B.1}$$

where

ρ_{CN}	is the cyanide contentration in the potassium cyanide solution (6.18.1) in milligrams per litre;
V_1	is the volume of silver nitrate solution (B.2.3) consumed, in millilitres;
$c(\mathrm{AgNO_3})$	is the concentration of silver nitrate solution in millimoles per litre;
M_{2CN}	is the molar mass of 2CN (= 52 g/mol);
V	is the volume of the potassium cyanide solution (6.18.1) in millilitres.