SLOVENSKI STANDARD
SIST ISO 6703-1:1996

01-junij-1996

Kakovost vode - Določanje cianida - 1. del: Določanje skupnega cianida

Water quality -- Determination of cyanide -- Part 1: Determination of total cyanide

Qualité de l'eau -- Dosage des cyanures -- Partie 1: Dosage des cyanures totaux

Ta slovenski standard je istoveten z: **ISO 6703-1:1984**

ICS:

13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances

SIST ISO 6703-1:1996 **en**

International Standard 6703/1

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Water quality — Determination of cyanide — Part 1: Determination of total cyanide

Qualité de l'eau — Dosage des cyanures — Partie 1: Dosage des cyanures totaux

First edition — 1984-09-01

UDC 543.2 : 546.267 **Ref. No. ISO 6703/1-1984 (E)**

Descriptors : water, quality, tests, determination, cyanides, water pollution.

Price based on 11 pages

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. Every 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6703/1 was prepared by Technical Committee ISO/TC 147, *Water quality*.

Printed in Switzerland

Contents

Page

Water quality — Determination of cyanide — Part 1: Determination of total cyanide

Attention is drawn to the toxicity of cyanide and to the need to take extreme care when handling cyanides and their solutions.

Carry out all operations in a fume cupboard. Avoid contact with the skin and eyes. When pipetting, always use a safety pipette (pipette by bulb). Detoxify samples and solutions containing cyanides or heavy metals in accordance with local official regulations.

Other chemicals specified in this part of ISO 6703 are also hazardous, for example pyridine.

0 Introduction

Cyanides may be present in water as hydrocyanic acid (prussic acid), as cyanide ions and as complex cyanides. They may be determined as total cyanide or as easily liberatable cyanide. If cyanide compounds are chlorinated, cyanogen chloride (CICN) is produced, and this compound has to be determined separately.

This International Standard comprises four parts as follows:

Part 1: Determination of total cyanide

Part 2: Determination of easily liberatable cyanide

Part 3: Determination of cyanogen chloride

Part 4: Determination of cyanide by diffusion at pH 6[1)]

The methods described in parts 1, 2 and 3 are suitable for controlling the quality of water and for the examination of municipal sewage and industrial effluents. They are appropriate to the technology available for the destruction of cyanides in treatment plants, and are based on the separation of liberated hydrogen cyanide (or in the case of ISO 6703/3, of cyanogen chloride) by stripping with a carrier gas.

The method specified in part 4 is suitable for the determination of smaller amounts of cyanide, depending on the concentrations of copper and nickel.

This part of ISO 6703 comprises four sections. Section one deals with the liberation and absorption of hydrogen cyanide. The other three sections deal with alternative methods for the quantitative determination of cyanide ions, as follows:

— photometric method with pyridine/barbituric acid (section two);

— titrimetric method using the Tyndall effect (section three);

— titrimetric method using an indicator (section four).

The specification of three alternative methods is necessary because each of the methods has its advantages and disadvantages. None can be quoted as applicable in all cases.

The applicability of each method is described in clauses 8, 16 and 24.

NOTE — Due to the different chemical behaviour of cyanide-containing or cyanide-producing substances, it is not possible to specify only one method for the quantitative determination of cyanide ions.

1 Scope and field of application

This part of ISO 6703 specifies three methods for the determination of total cyanide (see clause 2) in water.

The methods are applicable to water containing less than 100 mg of cyanide per litre, but higher concentrations may be determined by suitable dilution of the sample.

The methods and corresponding ranges of cyanide contents for which they are suitable are as follows:

— Photometric method with pyridine/barbituric acid : 0,002 to 0,025 mg;

— Titrimetric method using the Tyndall effect: > 0,005 mg;

— Titrimetric method using an indicator: > 0,05 mg.

1) At present at the stage of draft.

A large number of ions and compounds interfere with the determination. These are listed in the table 1, together with the concentrations below which they do not interfere (the list is not exhaustive). If present singly or in combination, up to the limiting concentrations, they do not interfere with the separation of hydrogen cyanide. The presence of aldehydes, e.g. formaldehyde, causes low cyanide values because of the formation of cyanohydrin.

If any of the limiting concentrations of the interferences is likely to be exceeded, the sample shall be diluted with distilled water before stabilization (see clause 6).

Other interference may arise from the presence of fatty acids, which will distil and form soaps during titration of alkaline solution, and from the presence of elementary sulfur.

Table 1 — Interferences

Interference	Limiting concentration, mg/l
Sulfide ions	1 000
Polysulfide ions	500
Sulfide and polysulfide ions	1 000
Sulfide ions	500
Thiosulfate ions	1 000
Thiocyanate ions	1 000
Carbonate ions	1 000
Cyanate ions	1 000
Nitrate ions	500
Nitrite ions	500
Ammonium ions	2 000
Iron(II) and iron(III) ions	5 000
Silver ions	50
Mercury ions	50
Chromate ions	300
Propionic acid	1 000
Phenol	1 000
Anthracene	100
Naphthalene	100
Anisaldehyde	10
Piperonal	10
Pyrrole	100
Pyridine	10
Chlorine (elemental)	250
Hydrogen peroxide	10
Perborate ions	10

2 Definition

For the purpose of this International Standard, the following definition applies.

total cyanide: Simple and complex bound cyanides including organic compounds containing cyanogen groups forming hydrogen cyanide under the conditions of this method. Cyanohydrins are detected in part. CN-groups of compounds defined as such may partly or completely form cyanide ions or hydrocyanic acid respectively in water. Mononitriles (R-CN), cyanate and thiocyanate ions and cyanogen chloride are not included.

Section one: Liberation and absorption of hydrogen cyanide

3 Principle

Heating the sample with hydrochloric acid in the presence of copper(I) ions. Entrainment of the liberated hydrogen cyanide in a current of air into an absorption vessel containing sodium hydroxide solution.

NOTES

1 Complex cobalt cyanides will not be determined quantitatively, because, according to their concentrations, they are decomposed to the extent of between 5 and 15 % only, this also applies to some organocyanide compounds.

2 The effect of the specified digestion procedure on cyanohydrine is not fully characterized.

4 Reagents

All reagents shall be of recognized analytical grade and the water used shall be distilled or deionized water.

4.1 Hydrochloric acid, solution, ϱ 1,12 g/ml.

4.2 Hydrochloric acid, solution, c(HCl) = 1 mol/l.

4.3 Sodium hydroxide, solution, c(NaOH) = 1 mol/l.

4.4 Sodium hydroxide, solution, c(NaOH) = 5 mol/l.

4.5 Tin(II) chloride, solution.[1)]

Dissolve 50 g of tin(II) chloride dihydrate ($SnCl_2.2H_2O$) in 40 ml of the hydrochloric acid solution (4.2) and dilute with water to 100 ml.

Prepare a fresh solution each week.

4.6 Phenolphthalein, solution, containing chloroform.

Dissolve 0,03 g of phenolphthalein in 90 ml of ethanol and add 10 ml of chloroform.

4.7 Zinc- and cadmium sulfate, solution.[1)]

Dissolve 100 g of zinc sulfate heptahydrate ($ZnSO_4.7H_2O$) and 100 g of cadmium sulfate octahydrate ($3CdSO_4.8H_2O$) in water and dilute with water to 1 000 ml.

4.8 Copper(II) sulfate, solution.

Dissolve 200 g of copper(II) sulfate pentahydrate ($CuSO_4.5H_2O$) in water and dilute with water to 1 000 ml.

4.9 Cadmium acetate, solution.[1)]

Dissolve 300 g of cadmium acetate dihydrate [$Cd(CH_3COO)_2.2H_2O$] in water and dilute with water to 1 000 ml.

4.10 Buffer solution, of pH 5,4.

Dissolve 6 g of sodium hydroxide (NaOH) in approximately 50 ml water, add 11,8 g of succinic acid ($C_4H_6O_4$) and dilute with water to 100 ml.

5 Apparatus

Usual laboratory equipment, and

5.1 Apparatus for the separation of hydrogen cyanide by stripping.

The apparatus shown in figure 1, or its equivalent, is recommended and comprises the following components.

5.1.1 Three-necked distillation flask, of capacity 500 ml, with standard conical joints (centre neck 29/32, side necks 14,5/23).

5.1.2 Reflux condenser (Liebig condenser).

5.1.3 Absorption vessels, protected against return of liquid.

5.1.4 Funnel.

5.1.5 Flowmeter.

5.1.6 Wash bottle, of capacity 250 ml, for purification of the air.

5.2 pH meter, with a glass electrode which will fit into the side necks of the distillation flask.

5.3 One-mark volumetric flasks, of capacities 25, 50, 250 and 1 000 ml.

6 Sampling and samples

If the sample contains undissolved cyanides, it is necessary to ensure homogeneous distribution of the undissolved substances in the sample and its dilutions. Immediately after sampling, add 5 ml of the sodium hydroxide solution (4.4), 10 ml of the phenolphthalein solution (4.6) and 5 ml of the tin(II) chloride solution (4.5) to each litre of sample or diluted

1) $SnCl_2$ is added as a reducing agent; zinc salt is added to provide stable zinc hexacyanoferrates, cadmium salts are added as sulfide acceptor and because of their bactericidal effect.