International Standard ISO 6703/3

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

Water quality — Determination of cyanide — Part 3: Determination of cyanogen chloride

Qualité de l'eau — Dosage des cyanures — Partie 3: Dosage du chlorure de cyanogène

First edition — 1984-09-01

UDC 543.2 : 546.267 **Ref. No. ISO 6703/3-1984 (E)**

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

Price based on 6 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 6703/3 was prepared by Technical Committee ISO/TC 147, *Water quality*.

Water quality — Determination of cyanide — Part 3: Determination of cyanogen chloride

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 (ClCN) 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 this part of ISO 6703, 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.

1 Scope and field of application

This part of ISO 6703 specifies a method for the determination of cyanides, as cyanogen chloride (see clause 2) in water.

The method is applicable for the determination of cyanogen chloride concentrations in the range 0,02 to 15 mg/l.

The ions and compounds listed in the table, if present singly or in combination at concentrations above the specified limiting concentration, interfere with the method (the list is not exhaustive).

The presence of aldehydes, e.g. formaldehyde, may give lower cyanide values because of the formation of cyanohydrin.

Table — Interferences

Interference	Limiting concentration mg/l
Sulfide ion	1 000
Polysulfide ion	300
Sulfide and polysulfide ion	1 000
Sulfite ion	500
Thiosulfate ion	1 000
Thiocyanate ion	1 000
Chlorine (elemental)	250

2 Definition

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

cyanogen chloride: The first reaction product when cyanide compounds are chlorinated.

Cyanogen chloride is a gas and is only slightly soluble in water, but is highly toxic even in low concentrations.

3 Principle

Addition of tin (II) chloride solution to the sample and entrainment of the cyanogen chloride liberated at pH 5,4 and room temperature by means of a current of air into an absorption solution containing pyridine/barbituric acid. Determination of the cyanogen chloride concentration photometrically.

1) At present at the stage of draft.

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 Tin(II) chloride, solution.

Dissolve 50 g of tin (II) chloride dihydrate ($SnCl_2 \cdot 2H_2O$) in the hydrochloric acid solution (4.2) and dilute with the same hydrochloric solution to 1 000 ml.

Prepare a fresh solution each week.

4.4 Sodium chloride, solution, c(NaCl) = 0,5 mol/l.

4.5 Sodium hydroxide, solution, c(NaOH) = 0,4 mol/l.

4.6 Pyridine/barbituric acid, solution.

Place 3 g of barbituric acid ($C_4H_4N_2O_3$) in a 50 ml one-mark volumetric flask, wash down the walls of the flask with just enough water to moisten the barbituric acid, add 15 ml of pyridine (C_5H_5N), and swirl to mix. Add 3 ml of the hydrochloric acid solution (4.1) and dilute to the mark with water.

Store overnight in a refrigerator and, if necessary, filter to eliminate any undissolved barbituric acid.

The solution is stable for 1 day if stored in the dark and for 1 week if stored in a refrigerator.

4.7 Potassium cyanide, standard solution corresponding to 10 mg of CN^- per litre.

Dissolve 25 mg of potassium cyanide in the sodium hydroxide solution (4.5) and dilute with the same sodium hydroxide solution to 1 000 ml in a one-mark volumetric flask.

Standardize this solution by titration with the silver nitrate solution (4.8), immediately before use or once each day, if numerous determinations are carried out.

4.8 Silver nitrate, solution, $c(AgNO_3)$ = 0,01 mol/l.

Store in a dark bottle.

4.9 Buffer solution, of pH 5,4.

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

4.10 Chloramine-T, solution.

Dissolve 0,5 g of chloramine-T trihydrate ($C_7H_7ClNaNSO_2 \cdot 3H_2O$) in water in a 50 ml one-mark volumetric flask and dilute to the mark with water.

Prepare fresh solution each week. Check each batch of chloramine-T against a calibration curve.

5 Apparatus

Usual laboratory equipment, and

5.1 Three-necked distillation flask, of capacity 500 ml, with standard conical joints (centre neck 29/32, side necks 14,5/23) and funnel, as shown in figure 1).

5.2 Absorption vessel, protected against return, with fritted glass G1 (see figure 2).

5.3 Separating funnel[1], of capacity 100 ml (see figure 3).

5.4 Sampling funnel[1], of capacity 10 ml (see figure 4).

5.5 Sampling funnel[1], of capacity 1 ml (see figure 5).

5.6 Volumetric flasks, of capacities 25, 50, 250 and 1 000 ml.

5.7 Flowmeter.

5.8 Spectrometer, with cells of optical path length 10 mm.

6 Sampling and samples

For expected cyanogen chloride concentrations of less than 0,15 mg/l, collect the sample in the separating funnel (5.3). For expected concentrations between 0,15 and 1,5 mg/l, collect the sample in the 10 ml sampling funnel (5.4) and for expected concentrations between 1,5 and 15 mg/l, collect the sample in the 1 ml sampling funnel (5.5).

Take the samples by immersing the appropriate funnels in the water to be sampled (with the stop-cocks closed below the surface of the water). If using the separating funnel (5.3), place 5 ml of the tin (II) chloride solution (4.3) in it before collecting the sample. Stopper the funnel immediately.

If one of the sampling funnels (5.4 or 5.5) was used to collect the sample, place 5 ml of the tin(II) chloride solution (4.3) in the separating funnel (5.3) and dilute with water to 100 ml.

Rinse the sampling funnel (5.4 or 5.5) with water and place it on the separating funnel (5.3). Open the stop-cock of the separating funnel first, then the lower, and finally the upper, stop-cock of the sampling funnel. After about 20 ml have run out of the separating funnel, close the stop-cocks of the separating funnel, remove the sampling funnel and stopper the separating funnel immediately.

Analyse as soon as possible and in any case within 24 h. If it is necessary to store the sample keep it cool and in the dark.

1) The exact volumes of the separating (5.3) and sampling funnels (5.4 and 5.5) should be determined prior to use.

Dimensions in millimetres

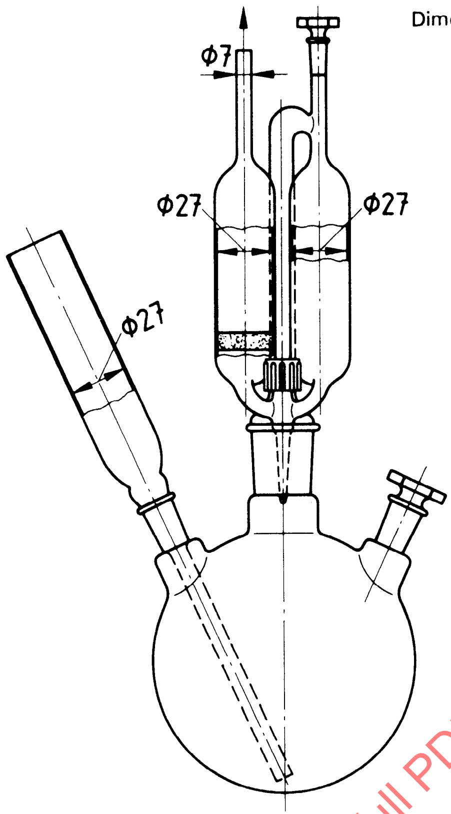

Figure 1 — Three-necked distillation flask

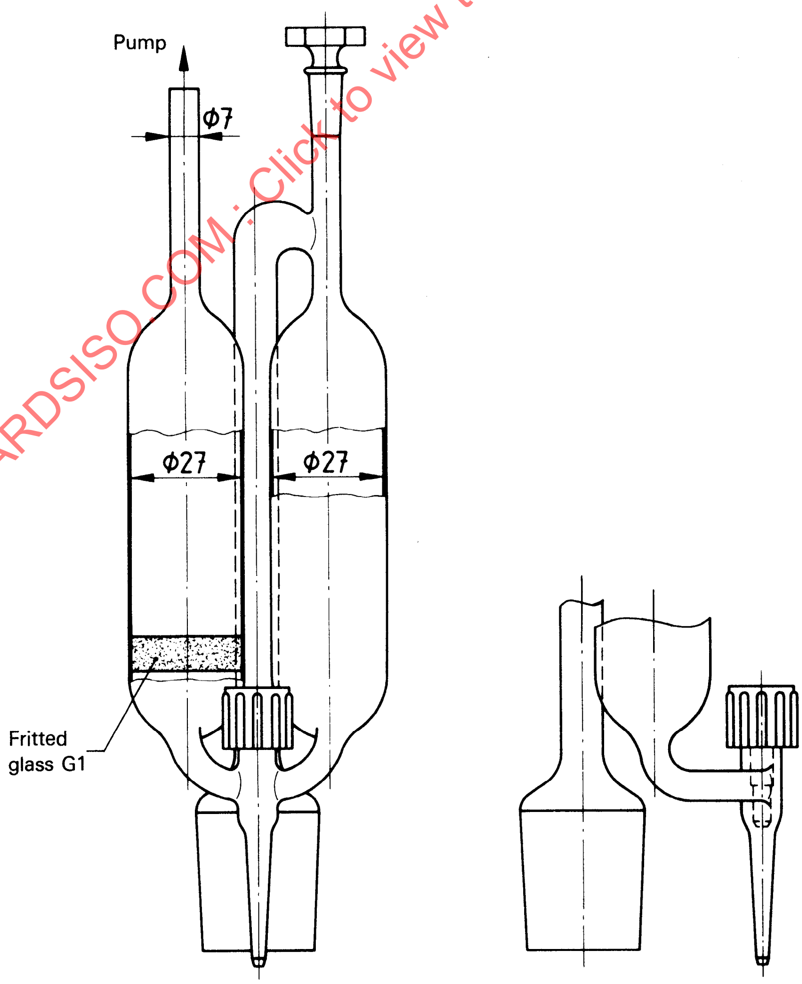

Figure 2 — Absorption vessel

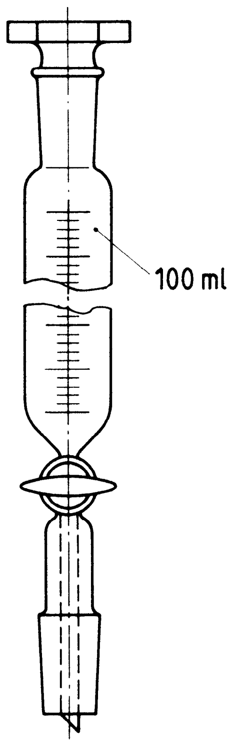

Figure 3 — Separating funnel

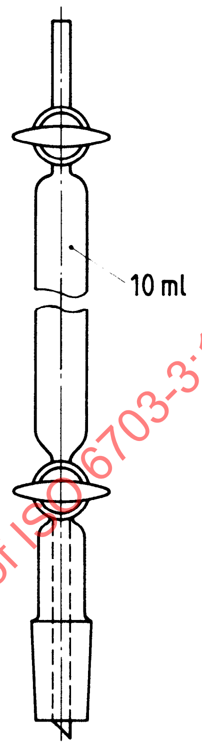

Figure 4 — 10 ml sampling funnel

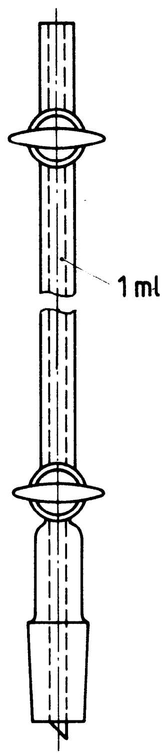

Figure 5 — 1 ml sampling funnel