

Water quality —

Part 2: Physical, chemical and biochemical methods —

Section 2.18 Methods for determination of easily liberatable cyanide

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Foreword

This Section of this British Standard, which has been prepared under the direction of the Environment and Pollution Standards Committee, is based on ISO 6703-2:1984 “*Water quality — Determination of cyanide — Part 2: Determination of easily liberatable cyanide*” but is not equivalent in technical content.

ISO 6703-2 was prepared by subcommittee 2, Physical, chemical and biochemical methods, of Technical Committee 147, Water quality, of the International Organization for Standardization (ISO) as a result of discussion in which the UK participated. For ease of publication, the text of ISO 6703-2 has been used as a basis for this Section of this British Standard and a number of amendments have been incorporated. The principal changes now incorporated are that the text has been amplified, to improve clarity and avoid misunderstandings in the following clauses: **1, 8, 12.2, 13, 16, 20, 21, 24, and 29**. In addition clause **8** now allows the use of smaller test portions in addition to dilution of absorption solutions with the sodium hydroxide solution.

This British Standard is being published in a series of Parts subdivided into Sections that will generally correspond to particular International Standards. Sections are being, or will be, published in Parts 1 to 6, which, together with Part 0, are listed below.

- *Part 0: Introduction;*
- *Part 1: Glossary;*
- *Part 2: Physical, chemical and biochemical methods;*
- *Part 3: Radiological methods;*
- *Part 4: Microbiological methods;*
- *Part 5: Biological methods;*
- *Part 6: Sampling.*

Terminology and conventions. As a result of using the ISO text some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

In British Standards it is current practice to use the symbol “L” for litre (and its submultiples) rather than “l” and to use the spelling “sulphur” etc., instead of “sulfur”, etc.

Additional information. If a high intensity beam of light is passed through a colloidal solution and the solution viewed at a right angle to the incident light, a scattering of light is observed. This is known as the Tyndall effect.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 12, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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 chemical specified in this Section of this British Standard 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.

There are 4 Sections in BS 6068 for the determination of cyanides in water as follows:

BS 6068-2.17:1986, *Methods for determination of total cyanide*.

BS 6068-2.18:1986, *Methods for determination of easily liberatable cyanide*.

BS 6068-2.19:1986, *Method for determination of cyanogen chloride*.

BS 6068-2.20:1986, *Method for determination of cyanide by diffusion at pH 6*.

The methods described in Sections 2.17, 2.18 and 2.19 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 BS 6068-2.19, of cyanogen chloride) by stripping with a carrier gas.

The method specified in BS 6068-2.20, using diffusion separation, is suitable for the determination of smaller amounts of cyanide, depending on the concentrations of copper and nickel.

This Section of this British Standard comprises 4 subsections. Subsection 1 deals with the liberation and absorption of hydrogen cyanide. The other 3 subsections deal with alternative methods for the quantitative determination of cyanide ions, as follows:

- photometric method with pyridine/barbituric acid (subsection 2);
- titrimetric method using the Tyndall effect (subsection 3);
- titrimetric method using an indicator (subsection 4).

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 Section of this British Standard specifies three methods for the determination of easily liberatable cyanide (see clause 2) in water.

The methods are applicable to water containing less than 50 mg of easily liberatable cyanide (as cyanide ions) per litre, and less than 100 mg of total cyanide (as cyanide ions) per litre, but higher concentrations may be determined by suitable dilution of the sample.

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

- Photometric method with pyridine/barbituric acid: applicable when the absorption solution contains 0,002 to 0,025 mg of cyanide, corresponding to 0,02 to 0,25 mg/l in a 100 ml aliquot portion of the undiluted sample;
- Titrimetric method using the Tyndall effect: applicable when the absorption solution contains > 0,005 mg of cyanide, corresponding to > 0,05 mg/l in a 100 ml aliquot portion of the undiluted sample;
- Titrimetric method using an indicator: applicable when the absorption solution contains > 0,05 mg of cyanide, corresponding to > 0,5 mg/l in a 100 ml aliquot portion of the undiluted sample.

A large number of ions and compounds interfere with the determination. These are listed in Table 1, together with the concentrations below which they do not interfere. If present singly or in combination, up to limiting concentrations, they do not interfere with the separation of hydrogen cyanide. The list is not exhaustive.

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
Copper(II) ions	100
Nickel(II) ions	50
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

If any of the limiting concentrations of the influences are likely to be exceeded, dilute the sample with distilled water before stabilization (see clause 6).

Prussiates (pentacyano complexes with iron), which cannot be destroyed by chlorination under normal conditions of waste water treatment, partly decompose (up to 50 %), releasing hydrocyanic acid under the conditions specified. If it is desired to exclude prussiates, the procedure specified in clause 6 and 7.1 has to be used. This procedure is only applicable, however, if the concentration of copper ions in the sample is less than 1 mg/l. The presence of aldehydes, e.g. formaldehyde, may give lower cyanide values because of the formation of cyanohydrin.

2 Definition

For the purpose of this Section of this British Standard the following definition applies.

easily liberatable cyanide

cyanide from substances with cyanide groups and a measurable hydrocyanic acid vapour pressure at pH 4 and room temperature

such substances include all cyanides which will undergo chlorination, especially hydrocyanic acid, alkali- and alkali earth metal cyanides, and complex cyanides of zinc, cadmium, silver, copper and nickel. Complex cyanides of iron and cobalt, nitriles, cyanates, thiocyanates and cyanogen chloride are not included