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Water quality — Determination of available weak and dissociable (WAD) cyanide —

Part 1: Method using ligand exchange, flow injection analysis (FIA), gas-diffusion and amperometric detection

Qualité de l'eau — Détermination du cyanure à acide faible dissociable (WAD) disponible —

Partie 1: Méthode par échange de ligand, analyse par injection en flux (FIA), diffusion gazeuse et détection ampérométrique

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

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Introduction

Methods using flow analysis automate wet chemical procedures are particularly suitable for the determination of many analytes in water in large sample series at a high analysis frequency.

Analyses can be performed by flow injection analysis (FIA) using the feature of an automatic dosage of the sample into a flow system (manifold) where the analytes in the sample react with the reagent solutions on their way through the manifold. The sample preparation can be integrated into the manifold. The reaction product is measured by a flow detector (e.g. amperometer).

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Water quality — Determination of available weak and dissociable (WAD) cyanide —

Part 1:
Method using ligand exchange, flow injection analysis (FIA), gas-diffusion and amperometric detection

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

1 Scope

This document specifies operationally defined methods for the determination of available WAD cyanide in various types of water such as drinking, ground, and surface, waters, and metallurgical processing tailings reclaim, heap leach barren, mill slurry tailings and leaching solutions, with cyanide concentrations from 5 µg/l to 2 000 mg/l expressed as cyanide ions in the undiluted sample. The range of application can be changed by varying the operation conditions, e.g. by using a different injection volume, thicker membrane, detector response, etc.

NOTE ISO 2080:2008, 3.105, defines free cyanide. The concentration of available WAD cyanide includes free cyanide and some of the metals complexed in solution as determined by a specified analytical method but not all of the metal complexes present in total cyanide (3,191).

In this method, six suitable mass concentration ranges from 5 µg/l to 50 µg/l, from 50 µg/l to 500 µg/l, from 0,5 mg/l to 5 mg/l, from 5 mg/l to 50 mg/l, from 50 mg/l to 500 mg/l and from 500 mg/l to 2 000 mg/l are described.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 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.

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ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
available WAD cyanide
sum of HCN, cyanide ions and cyanide bound in the metal-cyano complexes that are dissociated, using ligand reagents, if necessary, and sulfuric acid into HCN/CN^- in accordance with this document

4 Interferences

4.1 Interferences by oxidizing agents

Test for the presence of oxidizing agents, which can continue to oxidize the cyanide, leading to low results.

4.2 Interferences by sulfide

Sulfide will diffuse through the gas diffusion membrane and can be detected in the amperometric flow cell, causing high results. Oxidized products of sulfide can also rapidly convert CN^- to SCN^- at a high pH. A two-stage process is specified for sulfide removal. The initial stage should be carried out as soon as possible. Lead acetate test strips might not be sensitive enough to detect low levels of sulfide; therefore, treatment should be performed on samples where sulfide is suspected. Interference can be confirmed by analysing the sample with or without treatment.

If the measured cyanide in the untreated sample is significantly higher than in the treated sample, sulfide is likely present and treatment shall be performed to remove sulfide. In addition, the use of the sulfide removal and acidification reagent (6.8.4) is specified in this method. Its use will ensure removal of sulfide interference up to 50 mg/l of sulfide. This shall be applied within 24 h of taking the sample (see Clause 8).

NOTE USEPA method OIA-1677[7] uses of 0,1 mol/l HCl as acidification reagent, in the absence of sulfide in the samples.

5 Principle

The analytical procedure employed for determination of available WAD cyanide is divided into two parts: sample pre-treatment and cyanide detection. In the pre-treatment step, proprietary or non-proprietary ligand exchange reagents are added at room temperature to the sample, if needed to release the cyanide ions from mercury and nickel cyanide complexes. The ligand-exchange reagents form thermodynamically stable complexes with the transition metal ions, including: zinc, copper, cadmium, mercury, nickel and silver resulting in the release of bound cyanide ion from some metal-cyano complexes. Cyanide is not released from the more stable iron, gold and cobalt cyanide complexes.

The sample is treated with ligand exchange reagents, if necessary, and introduced into a carrier solution of the flow injection analysis (FIA) system via an injection valve and confluence downstream with a sulfuric acid solution containing sulfide removal reagent to measure available WAD cyanide. Ligand exchange reagents are needed to release cyanide from mercury cyanide and nickel cyanide complexes when the concentrations of mercury or nickel cyanides, relative to the weak and dissociable cyanide, will increase the result by more than 5 % relative. The released hydrogen cyanide (HCN) gas diffuses through a hydrophobic gas diffusion membrane into an alkaline acceptor stream where the CN^- is captured and sent to an amperometric flow cell detector with a silver-working electrode. In the presence of cyanide, silver electrode surface is oxidized at the applied potential (Eapp = 0,0 V vs. the reference electrode). The anodic current measured is proportional to the concentration of cyanide in the standard or sample injected.

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