SLOVENSKI STANDARD
SIST EN ISO 6878:2004

01-december-2004

Nadomeģ a:
SIST EN 1189:1997

Kakovost vode - Doloļ evanje fosforja - Spektrometrijska metoda z amonijevim molibdatom (ISO 6878:2004)

Water quality - Determination of phosphorus - Ammonium molybdate spectrometric method (ISO 6878:2004)

Wasserbeschaffenheit - Bestimmung von Phosphur - Photometrisches Verfahren mittels Ammoniummolybdat (ISO 6878:2004)

Qualité de l'eau - Dosage du phosphore - Méthode spectrométrique au molybdate d'ammonium (ISO 6878:2004)

Ta slovenski standard je istoveten z: **EN ISO 6878:2004**

ICS:

13.060.50	Preiskava vode na kemiļ ne snovi	Examination of water for chemical substances

SIST EN ISO 6878:2004 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 6878

June 2004

ICS 13.060.50

Supersedes EN 1189:1996

English version

Water quality - Determination of phosphorus - Ammonium molybdate spectrometric method (ISO 6878:2004)

Qualité de l'eau - Dosage du phosphore - Méthode spectrométrique au molybdate d'ammonium (ISO 6878:2004)

This European Standard was approved by CEN on 21 May 2004.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Foreword

This document (EN ISO 6878:2004) has been prepared by Technical Committee ISO/TC 147 "Water quality" in collaboration with Technical Committee CEN/TC 230 "Water analysis", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2004, and conflicting national standards shall be withdrawn at the latest by December 2004.

This document supersedes EN 1189:1996.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Endorsement notice

The text of ISO 6878:2004 has been approved by CEN as EN ISO 6878:2004 without any modifications.

INTERNATIONAL STANDARD

ISO 6878

Second edition
2004-06-01

Water quality — Determination of phosphorus — Ammonium molybdate spectrometric method

Qualité de l'eau — Dosage du phosphore — Méthode spectrométrique au molybdate d'ammonium

Reference number
ISO 6878:2004(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 6878 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

This second edition cancels and replaces the first edition (ISO 6878:1998), which has been technically revised.

Introduction

This International Standard specifies the determination of different forms of phosphorus compounds present in ground, surface and waste waters in various concentrations in the dissolved and undissolved state.

The user should be aware that particular problems could require the specification of additional marginal conditions.

Water quality — Determination of phosphorus — Ammonium molybdate spectrometric method

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard 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. It is absolutely essential that tests conducted according to this International Standard be carried out by suitably qualified staff. Molybdate and antimony waste solutions should be disposed of properly.

1 Scope

This International Standard specifies methods for the determination of

— orthophosphate (see Clause 4);

— orthophosphate after solvent extraction (see Clause 5);

— hydrolysable phosphate plus orthophosphate (see Clause 6);

— total phosphorus after decomposition (see Clauses 7 and 8).

The methods are applicable to all kinds of water including seawater and effluents. Phosphorus concentrations within the range of 0,005 mg/l to 0,8 mg/l may be determined in such samples without dilution.

A solvent extraction procedure allows smaller phosphorus concentrations to be determined with a detection limit of about 0,000 5 mg/l.

2 Interferences

See Annex A for some known interferences. There may be others and it is recommended to verify whether any such interferences exist and take action to eliminate them.

3 Principle

Reaction of orthophosphate ions with an acid solution containing molybdate and antimony ions to form an antimony phosphomolybdate complex.

Reduction of the complex with ascorbic acid to form a strongly coloured molybdenum blue complex. Measurement of the absorbance of this complex to determine the concentration of orthophosphate present.

Polyphosphate and some organophosphorus compounds are determined if converted to molybdate reactive orthophosphate formed by sulfuric acid hydrolysis.

Many organophosphorus compounds are converted to orthophosphate by mineralization with peroxodisulfate. Nitric acid-sulfuric acid mineralization is used if a more vigorous treatment is required.

4 Determination of orthophosphate

4.1 Reagents

During the analysis, use only reagents of recognized analytical grade and only water having a phosphate content that is negligible compared with the lowest concentration to be determined in the samples.

For low phosphate contents, double-distilled water from an all-glass apparatus is recommended.

4.1.1 Sulfuric acid solution, $c(H_2SO_4) \approx 9$ mol/l.

Add 500 ml ± 5 ml of water to a 2 l beaker. Cautiously add, with continuous stirring and cooling, 500 ml ± 5 ml of sulfuric acid, $\rho = 1{,}84$ g/ml. Mix well and allow the solution to cool to room temperature.

4.1.2 Sulfuric acid solution, $c(H_2SO_4) \approx 4{,}5$ mol/l.

Add 500 ml ± 5 ml of water to a 2 l beaker. Cautiously add, with continuous stirring and cooling, 500 ml ± 5 ml of sulfuric acid (4.1.1). Mix well and allow to cool to room temperature.

4.1.3 Sulfuric acid solution, $c(H_2SO_4) \approx 2$ mol/l.

Add 300 ml ± 3 ml of water to a 1 l beaker. Cautiously add 110 ml ± 2 ml of sulfuric acid solution (4.1.1), with continuous stirring and cooling. In a measuring flask, dilute to 500 ml ± 2 ml with water and mix well.

4.1.4 Sodium hydroxide solution, $c(NaOH) = 2$ mol/l.

Dissolve 80 g ± 1 g of sodium hydroxide pellets in water, cool and dilute to 1 l with water.

4.1.5 Ascorbic acid solution, $\rho = 100$ g/l.

Dissolve 10 g ± 0,5 g of ascorbic acid ($C_6H_8O_6$) in 100 ml ± 5 ml water.

NOTE The solution is stable for 2 weeks if stored in an amber glass bottle in a refrigerator and can be used as long as it remains colourless.

4.1.6 Acid molybdate, Solution I.

Dissolve 13 g ± 0,5 g of ammonium heptamolybdate tetrahydrate [$(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$] in 100 ml ± 5 ml of water. Dissolve 0,35 g ± 0,05 g of antimony potassium tartrate hemihydrate [$K(SbO)C_4H_4O_6{\cdot}½H_2O$] in 100 ml ± 5 ml of water.

Add the molybdate solution to 300 ml ± 5 ml of sulfuric acid (4.1.1) with continuous stirring. Add the tartrate solution and mix well.

NOTE The reagent is stable for at least 2 months if stored in an amber glass bottle.

4.1.7 Acid molybdate, Solution II.

Cautiously add 230 ml ± 0,5 ml of sulfuric acid (4.1.1) to 70 ml ± 5 ml of water, cool. Dissolve 13 g ± 0,5 g of ammonium heptamolybdate tetrahydrate [$(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$] in 100 ml ± 5 ml of water. Add to the acid solution and mix well. Dissolve 0,35 g ± 0,05 g of antimony potassium tartrate hemihydrate [$K(SbO)C_4H_4O_6{\cdot}½H_2O$] in 100 ml ± 5 ml of water. Add to the molybdate-acid solution and mix well.

This reagent is used when the sample is acidified with sulfuric acid (4.1.2) (see also Clauses 6, 7 and 8).

NOTE The reagent is stable for at least 2 months if stored in an amber glass bottle.