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VEE KVALITEET. ORTOFOSFAADI JA ÜLDFOSFORI SISALDUSE MÄÄRAMINE VOOLUANALÜÜSIL (FIA JA CFA). OSA 2: PIDEVVOOLUANALÜSAATORI MEETOD (CFA)

Water quality - Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA) - Part 2: Method by continuous flow analysis (CFA) (ISO 15681-2:2018)



EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

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Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas. Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 05.12.2018.		
Standard on kättesaadav Eesti Standardimis- ja Akrediteerimiskeskusest.	05.12.2018. The standard is available from the Estonian Centre for Standardisation and Accreditation.	
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ICS 13.060.50

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EUROPEAN STANDARD NORME EUROPÉENNE **EUROPÄISCHE NORM**

EN ISO 15681-2

December 2018

ICS 13.060.50

Supersedes EN ISO 15681-2:2004

English Version

Water quality - Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA) - Part 2: Method by continuous flow analysis (CFA) (ISO 15681-2:2018)

Qualité de l'eau - Dosage des orthophosphates et du phosphore total par analyse en flux (FIA et CFA) -Partie 2: Méthode par analyse en flux continu (CFA) (ISO 15681-2:2018)

Wasserbeschaffenheit - Bestimmung von Orthophosphat und Gesamtphosphor mittels Fließanalytik (FIA und CFA) - Teil 2: Verfahren mittels kontinuierlicher Durchflussanalyse (CFA) (ISO 15681-2:2018)

This European Standard was approved by CEN on 10 August 2018.

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CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

European foreword

This document (EN ISO 15681-2:2018) 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of an identical text or by endorsement, at the latest by June 2019, and conflicting national standards shall be withdrawn at the latest by June 2019.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN ISO 15681-2:2004.

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

Endorsement notice

The text of ISO 15681-2:2018 has been approved by CEN as EN ISO 15681-2:2018 without any modification.

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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 of 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 <u>www.iso</u> .org/iso/foreword.html.

This document 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 15681-2:2003), which has been technically revised. The main changes compared to the previous edition are as follows:

- a) the reagents have been adjusted to decrease the pH to enhance the colour reaction;
- b) the figures in <u>Annex A</u> have been revised.

A list of all parts in the ISO 15681 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at <u>www.iso.org/members.html</u>.

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Introduction

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

Analysis can be performed by flow injection analysis (FIA)^{[6][8]} or continuous flow analysis (CFA)^[9]. Both methods share the feature of an automatic dosage of the sample into a flow system (manifold) where the analyte in the sample reacts with the reagent solutions on its way through the manifold. The sample preparation may be integrated in the manifold. The amount of reaction product is measured in a flow detector (e.g. flow photometer). This document describes the CFA method.

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

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Water quality — Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA) —

Part 2: Method by continuous flow analysis (CFA)

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 continuous flow analysis (CFA) methods for the determination of orthophosphate in the mass concentration range from 0,01 mg/l to 1,00 mg/l P, and total phosphorus in the mass concentration range from 0,10 mg/l to 10,0 mg/l P. The method includes the digestion of organic phosphorus compounds and the hydrolysis of inorganic polyphosphate compounds, performed either manually, as described in ISO 6878 and in References [4], [5] and [7], or with an integrated ultraviolet (UV) digestion and hydrolysis unit.

This document is applicable to various types of water, such as ground, drinking, surface, leachate and waste water. The range of application can be changed by varying the operating conditions.

This method is also applicable to the analysis of seawater, but with changes in sensitivity by adapting the carrier and calibration solutions to the salinity of the samples.

It is also applicable to analysis using 10 mm to 50 mm cuvettes depending on the desired range. For extreme sensitivity, 250 mm and 500 mm long way capillary flow cells (LCFCs) can be used. However, the method is not validated for these two uses. Changes in sensitivity and calibration solutions could be required.

<u>Annex A</u> provides examples of a CFA system. <u>Annex B</u> gives performance data from interlaboratory trials. <u>Annex C</u> gives information of determining orthophosphate-P and total-P by CFA and tin(II) chloride reduction.

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 5667-1, Water quality — Sampling — Part 1: Guidance on the design of sampling programmes and sampling techniques

ISO 5667-3:2018, Water quality — Sampling — Part 3: Preservation and handling of water samples

ISO 6878:2004, Water quality — Determination of phosphorus — Ammonium molybdate spectrometric method

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

No terms and definitions are listed in this document.

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/

4 Interferences

4.1 General interferences

Refer to ISO 6878:2004, Annex A for a list of general interferences. In addition, or contrary to the cited standard, the following applies:

- a) arsenate causes serious interference: 100 μ g/l As, present as arsenate, results in a response comparable to approximately 30 μ g/l P;
- b) if the silicate concentration in samples is not greater than 60 times the phosphorus concentration, interferences by silicate can be neglected;
- c) fluoride interference is significant above 50 mg/l;
- d) nitrite interference is significant above 5 mg/l; the interference can be eliminated by acidifying samples after collection;
- e) for samples containing high concentrations of oxidizing agents, the amount of added reduction reagent can be insufficient; in this case, remove the oxidizing material prior to digestion;
- f) the self-absorption of the sample can be compensated for by measuring, in addition to the sample signal (9.6), the signal of the sample without the admixture of the reagents; in this case, the difference of the two responses is used for the evaluation (Clause 10).

4.2 Interferences in the determination of total-P

Samples containing solids or suspended particles can show low values when analysed by the UV method, if the particles are not completely transported into the UV unit. The error can be minimized by stirring the sample immediately before or during sampling, in order to ensure that a representative sample is delivered into the analyser, and by reducing the particle size.

The interferences from silicate, nitrite, fluoride and iron described for the orthophosphate determination are generally not observed in the UV method, due to the pre-digestion and the higher analytical range.

The efficiency of the UV digestion can be affected for water samples with chemical oxygen demand (COD) values of more than 10 times the highest concentrations of the calibration solutions (6.22). In this case, the sample should be diluted.