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Water quality — Determination of selected parameters by discrete analysis systems —

Part 1:
Ammonium, nitrate, nitrite, chloride, orthophosphate, sulfate and silicate with photometric detection

Qualité de l'eau — Détermination de paramètres sélectionnés par des systèmes d'analyse discrète —

Partie 1: Ammonium, nitrate, nitrite, chlorure, orthophosphate, sulfate et silicate par détection photométrique

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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. www.iso.org/directives

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

The committee responsible for this document is ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

ISO 15923 consists of the following parts, under the general title *Water quality — Determination of selected parameters by discrete analysis systems*:

— *Part 1: Ammonium, nitrate, nitrite, chloride, orthophosphate, sulfate and silicate with photometric detection*

Introduction

Many photometric determinations can be automated with a discrete analysis system. With one single apparatus, a large number of different parameters can be determined, and the parameters to be determined can be specified for each sample. Working with small volumes requires less sample material and reagent.

Samples that fall beyond the normal measuring range can either be automatically diluted or measured again with a different measuring range.

This part of ISO 15923 specifies methods for the automatic determination of ammonium, nitrate, nitrite, chloride, orthophosphate, and silicate with photometric detection and a turbidimetric determination of sulfate using a discrete analysis system. The field of application is water (ground, potable, surface, waste, eluates, and boiler water).

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Water quality — Determination of selected parameters by discrete analysis systems —

Part 1:
Ammonium, nitrate, nitrite, chloride, orthophosphate, sulfate and silicate with photometric detection

WARNING — Persons using this part of ISO 15923 should be familiar with normal laboratory practice. This part of ISO 15923 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.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this part of ISO 15923 be carried out by suitably qualified staff.

1 Scope

This part of ISO 15923 specifies methods for the automatic performance of spectrophotometric and turbidimetric analyses with a discrete analysis system for determining ammonium, nitrate, nitrite, chloride, orthophosphate, sulfate, and silicate. The field of application is ground, potable, surface, waste, eluates, and boiler water.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 Principle

A discrete analysis system is an automated system for spectrophotometric and turbidimetric determinations.

The colour reactions take place in reaction cells, which may be cuvettes, in an incubator. For each determination, a separate reaction cell is used. Preset volumes of the sample and the reagents are pipetted into the cells and mixed.

After expiry of the incubation period, the absorbance of the solution is measured at the wavelength applicable to the determination. This is done by passing the cuvettes through the photometer or by transferring the measuring solution from the reaction cells to a photometer with a flow-through cell.

4 Interferences

Particles present in the sample can lead to blockages and will interfere with the photometric measurement. Filtration of all samples through a 0,45 µm membrane filter is recommended, except for the determination of total phosphate and Kjeldahl nitrogen digests (see Annexes B and F). Particles can also be removed by settlement, centrifugation, or dialysis.

This method is applicable to samples in a pH range from 5 to 9, which covers most natural waters. Samples outside this range may require pH correction.

Inherent colour or turbidity of the sample interferes with the analysis. On the prevention of such interference, see Annex A. Interferences specific to each parameter are discussed in Annexes B to H.

NOTE   Interference by inherent colour shall be compensated by measuring the absorbance of the sample before the addition of the chromogenic reagent (sample blanking) or by making use of a compensating solution (measuring solution without a chromogenic compound). For further details, see Annex A. A safe procedure for the correction of turbidity cannot really be given. The Lambert-Beer law does not apply to turbid solutions. Furthermore, many chromogenic reagents and coloured complexes are adsorbed on particles.

5 Reagents

Reagents for each parameter are specified in Annexes B to H. Use only reagents of recognized analytical grade, unless otherwise specified in the relevant annex. Dry all solid reagents for at least 1 h at (105 ± 5) °C, provided that they are thermally stable. Store the dried solid in a desiccator before weighing. Reagent volumes specified in Annexes B to H may be adjusted to suit local requirements or different instrument specifications.

5.1   Water, complying with grade 1 as defined in ISO 3696.

6 Apparatus

6.1   Discrete analysis system, generally consisting of the following components:

6.1.1   Sample injection device, for automated or manual operation.

6.1.2   Sample container.

6.1.3   Reagent container, refrigerated or not.

6.1.4   Incubator with temperature control, capable of maintaining a constant temperature of e.g. 37 °C.

6.1.5   UV/VIS detector, e.g. spectrophotometer, suitable for a wavelength range usually between 340 nm and 880 nm.

6.1.6   Control and data processing unit.

6.1.7   Recording device, e.g. PC with software for data acquisition and evaluation.

7 Sampling and sample preparation

Use clean vessels for sampling.

Turbidity or particulates interfere with spectrophotometric detection. Clarify samples by filtration through a 0,45 µm membrane (settlement, centrifugation, or dialysis may also be used). To avoid

contamination by the filter membrane, discard the first 20 ml to 30 ml of filtrate. Samples for the determination of total phosphate should not be filtered. Refer to Annex F.

Prepare and store the sample in accordance with Annexes B to H or with ISO 5667-3 if no specific guidance is given in the relevant annex.

Prepare a sample of water (5.1) in the same way as the sample, to be used as a blank.

Prepare a control standard solution from the primary control standard containing a level of analyte similar to the samples. Run as a sample at appropriate intervals in the batch, according to local requirements. A minimum interval of once every 20 samples is recommended.

8 Calibration

8.1 Calibration function

When the analytical system is first evaluated and at intervals afterwards, establish a calibration function for each parameter (see ISO 8466-1 or ISO 8466-2) as follows:

Using the primary calibration standard, prepare an appropriate series of calibration solutions for the respective parameter as described in Annexes B to H, including a zero-concentration solution.

Analyse the calibration solutions according to Clause 9 and the instrument manufacturer's instructions.

Confirm the validity of the data obtained and use to calculate the regression line as specified in ISO 8466-1 or ISO 8466-2.

Verify the continuing validity of the established calibration function by analysing an appropriate calibration standard solution, at regular intervals according to the accuracy requirements or at least at the end of the batch.

Recalibrate if necessary.

8.2 Calibration validity check

If the full calibration function is not established daily, carry out an initial calibration validity check by analysing two calibration standard solutions in the lower and upper third of the calibrated working range after the setup procedure (see Clause 9).

Verify the continuing validity of the established calibration function by analysing an appropriate calibration standard solution at regular intervals according to the accuracy requirements or at least at the end of the batch.

Recalibrate if necessary.

9 Procedure

Set up the discrete analysis system according to the instrument manufacturer's instructions.

Calibrate the system according to Clause 8 and the instrument manufacturer's instructions.

Prepare the samples according to Clause 7 and Annexes B to H. A consistent incubation temperature and time are essential for the stability of the absorbance measurements. For guidance on recommended incubation temperatures and times, refer to Annexes B to H. Note that the incubation times given in Annexes B to H are recommendations, which may be varied according to experience.

Measure the absorbance of the samples using the conditions in Annexes B to H and the instrument manufacturer's instructions. Measure the blank according to Annex A and the instrument manufacturer's instructions.

If the absorbance of the sample exceeds that of the top calibration solution, dilute the sample, or reduce the sample intake by an appropriate factor to bring it into the upper half of the calibration range, and reanalyse. If necessary, blank correct the sample absorbances (see Annex A).

The procedures in Annexes B to H may be modified for different instruments or to change the range or sensitivity of the method for different parameter concentrations or sample types.

10 Calculation

Calculate the mass concentration, ρ, of the parameter in question in micrograms per litre (µg/l) or milligrams per litre (mg/l) from the calibration line (see Clause 8), using the corrected absorbance values obtained (see Clause 9), as specified in ISO 8466-1 or ISO 8466-2. Take account of any dilution factors. This calculation can usually be carried out automatically using the instrument software.

11 Expression of results

Results shall be expressed to a maximum of three significant figures.

EXAMPLES   Phosphate = 1,11 mg/l P (3 sig fig), 1,1 mg/l P (2 sig fig), 1 mg/l (1 sig fig).

Before reporting results, it is important to determine the required units of expression. For example, ammonium, nitrate, and nitrite can be expressed as N or as the relevant ion. Results for orthophosphate can be expressed as P or PO_4, and silicate may be expressed as SiO_4, SiO_2, or Si.

Appropriate conversion factors are given in Table 1.

Table 1 — Conversion factors

Parameter	Units	Conversion factor	Converted units
Ammonia	mg/l N	1,286	mg/l NH_4
Ammonia	mg/l NH_4	0,777 8	mg/l N
Nitrate	mg/l N	4,427	mg/l NO_3
Nitrate	mg/l NO_3	0,225 9	mg/l N
Nitrite	mg/l N	3,285	mg/l NO_2
Nitrite	mg/l NO_2	0,304 4	mg/l N
Orthophosphate	mg/l P	3,066	mg/l PO_4
Orthophosphate	mg/l PO_4	0,326 1	mg/l P
Silicate	mg/l Si	3,279	mg/l SiO_4
Silicate	mg/l SiO_4	0,305 0	mg/l Si
Silicate	mg/l Si	2,139	mg/l SiO_2
Silicate	mg/l SiO_2	0,467 4	mg/l Si
Silicate	mg/l SiO_2	1,533	mg/l SiO_4
Silicate	mg/l SiO_4	0,652 5	mg/l SiO_2

12 Test report

The test report shall contain at least the following information:

a) the test method used, together with a reference to this part of ISO 15923 (i.e. ISO 15923-1:2013);

b) the details required for identification of the sample;

c) the date of the analysis;

d) the analytical results (see Clause 11);

e) any deviation from this method and a report of circumstances that may have affected the results.