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

Part 2: Chromium(VI), fluoride, total alkalinity, total hardness, calcium, magnesium, iron, iron(II), manganese and aluminium with photometric detection

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

Partie 2: Chrom(VI), fluorure, alcalinité totale, dureté totale, calcium, magnésium, fer, (fer(II), manganèse et aluminium avec 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 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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

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*.

A list of all parts in the ISO 15923 series can be found on the ISO website.

Introduction

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

Samples that fall outside the normal range of measurement can either be automatically diluted or analysed using a different measuring range.

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

Part 2: Chromium(VI), fluoride, total alkalinity, total hardness, calcium, magnesium, iron, iron(II), manganese and aluminium with photometric 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 methods for the automatic determination of chromium(VI), fluoride, total alkalinity, total hardness, calcium, magnesium, iron, iron(II), manganese and aluminium with photometric determination using a discrete analysis system. The field of application is water (ground, potable, surface, waste, eluates and boiler water). The method can also be applied to marine waters with matrix matching of standard and control solutions. Note that some parameters, notably iron, manganese and aluminium and possibly chromium(VI), calcium and magnesium may not be completely quantified if the sample contains particulates. Samples can be digested in acid, as long as the buffering capacity of the reaction mixture is not exceeded. Such procedures are beyond the scope of this document, which is best suited to the determination of dissolved metals following on-site filtration.

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 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 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. Pre-set volumes of the sample and the reagents are pipetted into the cells and mixed.

Following the incubation period, the absorbance of the solution is measured at the wavelength applicable to the determination. Depending on which instrument is used, measurement is achieved 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.

The specific chemistry for each parameter is given in the relevant annex.

5 Interferences

Particles present in the sample can lead to blockages and will interfere with the photometric measurement. Filtration of samples containing particles through a 0,45 µm membrane filter is recommended. Particles can also be removed by settlement, centrifugation or dialysis.

If the sample is filtered prior to analysis, the fraction of any of the parameters that is adsorbed onto the surface of particles will not be measured.

Inherent colour or turbidity of the sample can interfere with the analysis. Two possible procedures to correct for any inherent colour are described in Annex A.

A reliable procedure for the correction of turbidity cannot really be given. The Beer-Lambert law does not apply to turbid solutions. Furthermore, many chromogenic reagents and coloured complexes are adsorbed on particles.

Interferences specific to each parameter are discussed in the relevant annex.

6 Reagents

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

For many of the reagents, calibration and control standards specified in this document, commercial preparations are available and it is quite acceptable to use them provided that manufacturer's instructions relating to storage and stability are followed.

6.1 Water, complying with the specification for grade 1 as defined in ISO 3696.

7 Apparatus

7.1 Discrete analysis system, generally consisting of the following components.

7.1.1 Sample injection device, for automated or manual operation.

7.1.2 Sample container.

7.1.3 Reagent container, refrigerated or not.

7.1.4 Incubator with temperature control, capable of maintaining a constant temperature.

7.1.5 Visible wavelength detector, e.g. spectrophotometer, suitable for a wavelength range usually between 400 nm and 880 nm.

7.1.6 Control and data processing unit.

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

7.2 Routine laboratory apparatus, including

7.2.1 Balance, capable of measuring to 0,000 1 g.

7.2.2 Oven.

7.2.3 Exsiccator.

7.2.4 Glassware, including volumetric flasks and beakers.

7.2.5 Autopipettes, capable of dispensing volumes from 50 µl to 500 µl.

8 Sampling and sample preparation

Use clean vessels for sampling.

Turbidity or particulates interfere with spectrophotometric detection. Using an appropriate filtration apparatus, clarify any samples containing particles by filtering through a 0,45 µm membrane (settlement, centrifugation or dialysis may also be used). To avoid contamination by the filter membrane, discard the first few millilitres of filtrate.

ISO 5667-3 offers guidance on the preparation and storage of samples. However, the stability of some of the parameters covered by this document may vary according to conditions such as the pH and other constituents present in the sample. Stability trials should be carried out locally for each matrix type. The guidance in ISO 5667-3 for preservation of samples for iron, manganese, aluminium, calcium and magnesium recommends acidifying the sample to between pH 1 and 2, but this may not be appropriate for discrete analysis methods where pH is critical, e.g. calcium and manganese. In such cases, it is important to ensure that the buffering capacity of the reaction mixture is not exceeded. Fluoride is stable for at least one month with no pre-treatment. For chromium(VI), best practice is to analyse the sample as soon as possible after sampling. ISO 18412[2] recommends a maximum of 4 d refrigerated storage, but ISO 23913[3] specifies storage for no more than 24 h at 2 °C to 5 °C.

Prepare a sample of water (6.1) 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 it as a sample at appropriate intervals in the batch, according to local requirements. A minimum interval of once every 20 samples is recommended. Instructions for preparing a primary control standard are given in Annexes B to J.

9 Calibration

9.1 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 (Annexes B to J). Use water (6.1) as a zero concentration calibration 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.

During the analysis, verify the continuing validity of the established calibration function by analysing an appropriate calibration standard solution, at regular intervals according to local accuracy requirements, or at least at the end of the batch. Recalibrate, if necessary. It is recommended that calibration verification is carried out using a calibration solution in the upper third of the calibration range.

9.2 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 (see Clause 10).

Verify the continuing validity of the established calibration function as described in 9.1.

10 Procedure

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

Calibrate the system according to Clause 9 and the instrument manufacturer's instructions.

Prepare the samples according to Clause 8 and Annexes B to J. A consistent incubation temperature and time are essential for the stability of the absorbance measurements. Measure the absorbance of the samples using the instrument manufacturer's recommended instructions. Measure the blank according to Annex A and the instrument manufacturer's instructions.

If the absorbance of a sample exceeds that of the top calibration solution, dilute the sample using water (6.1), or reduce the sample intake by an appropriate factor to bring it into the upper half of the calibration range, and reanalyse. If necessary, subtract the absorbance of the blank from that of the samples (see Annex A).

The analytical procedure may be modified for different instruments, or to change the range or sensitivity of the method for different parameter concentrations or sample types.

11 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 9), using the corrected absorbance values obtained (see Clause 10), 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.

12 Expression of results

Results shall be expressed as µg/l or mg/l to a maximum of three significant figures.

EXAMPLES A reading of 11,12 µg/l, rounding to: 11,1 µg/l (3 sig fig), 11 µg/l (2 sig fig), 10 µg/l (1 sig fig).

13 Test report

This test report shall contain at least the following information:

a) the test method used, together with a reference to this document, i.e. ISO/TS 15923-2:2017;

b) the details required for identification of the sample;

c) the date of the analysis;

d) the analytical results (see Clause 12);

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

Annex A
(normative)

Correction for inherent colour

A.1 General

Correction for any inherent colour in the sample is necessary. Two possible procedures are described in A.2 and A.3. Accurate correction for turbidity is not possible using these methods because the Beer-Lambert law does not apply. Discrete analysis systems can be programmed to carry out colour correction automatically.

A.2 Sample blanking

The blank absorption measurement is done after dispensing the sample and, if applicable, one or more reagents that could produce a colour change in the sample (for example because of the influence of the pH), but before dispensing the chromogenic reagent. This blank value is adjusted to take account of the ratio between the sample volumes with and without the chromogenic reagent, and subtracted from the final absorption measurement. The standards are measured in the same way.

A.3 Use of a compensating solution

When using a compensating solution, a second measuring solution is prepared using the same volumes of sample and reagent, in which the compound responsible for forming the colour is omitted. This can be achieved either by adding an equal volume of water (6.1) instead of the chromogenic reagent, or by preparing a separate reagent from which the chromogenic compound is omitted. The absorption of the compensating solution is deducted from the absorption of the sample solution.