
**Water quality — Determination of
sulfates — Method by continuous flow
analysis (CFA)**

*Qualité de l'eau — Dosage des sulfates — Méthode par analyse en flux
continu (CFA)*

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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.

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 22743 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 and 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) [1], [2] or by continuous flow analysis (CFA) [3]. Both methods share 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 its way through the manifold. The sample preparation may be integrated in the manifold. The reaction product is measured in a flow detector (e.g. flow photometer). The detector produces a signal from which the concentration of the parameter is calculated.

It is necessary to examine whether and to what extent particular problems will require the specification of additional marginal conditions.

In this International Standard, only the CFA procedure is described.

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Water quality — Determination of sulfates — Method by continuous flow analysis (CFA)

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

IMPORTANT — It is absolutely essential that tests conducted according to this standard be carried out by suitably trained staff.

1 Scope

This International Standard specifies a continuous flow analysis (CFA) method for the determination of sulfate in various types of water (such as ground water, drinking water and waste water).

The method is applicable to samples with a sulfate (SO_4) mass concentration from 30 mg/l to 300 mg/l. Other concentration ranges are applicable, provided they cover exactly one decade of concentration units (e.g. 100 mg/l to 1 000 mg/l).

2 Normative references

[ISO 22743:2006](https://standards.iteh.ai/catalog/standards/sist/d2e041b0-1b12-4d7c-8459-3f490491c1ac/iso-22743-2006)

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The following referenced documents are indispensable for the application 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: Guidance on the 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 Interferences

Calcium-, magnesium-, iron- and aluminium ions interfere with the determination of sulfate (negative bias). Treatment with a cation exchange resin will reduce the interferences.

Sulfide, sulfite, phosphate and tannic acids may lead to a positive bias, but concentrations of these substances which cause measurable interferences are usually not found in real samples.

4 Principle

Sulfate reacts in an acidic solution with barium chloride to form barium sulfate. The excessive barium chloride reacts with methylthymol blue in an alkaline solution to a chelated barium methylthymol blue. The absorbance of the free non-chelated methylthymol blue is measured at $460 \text{ nm} \pm 10 \text{ nm}$ in a flow through detector. Other metals, such as calcium, magnesium, iron and aluminium, are removed by an ion exchange column prior to the reactions with barium.

5 Reagents

Use analytical grade chemicals, unless otherwise specified, and check the blank value of the reagents.

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

5.2 Barium chloride dihydrate, $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$.

5.3 Concentrated hydrochloric acid, $c(\text{HCl}) = 12 \text{ mol/l}$, $\rho(\text{HCl}) = 1,19 \text{ g/ml}$.

5.4 Methylthymol blue, $(\text{C}_{37}\text{H}_{40}\text{N}_2\text{O}_{13}\text{SNa}_4 \text{ or } \text{C}_{37}\text{H}_{43}\text{N}_2\text{O}_{13}\text{SNa})$.

5.5 Surfactant, polyoxyethylene-23-laurylether ($\text{C}_{12}\text{H}_{25}(\text{OCH}_2\text{CH}_2)_{23}\text{OH}$), a mass fraction of 30 % solution in water.

5.6 Sodium hydroxide, (NaOH) .

5.7 Ethylenedinitrilotetracetic acid tetrasodium salt, $\text{Na}_4\text{-EDTA}$, $(\text{C}_{10}\text{H}_{12}\text{N}_2\text{Na}_4\text{O}_8)$.

5.8 Ammonium chloride, (NH_4Cl) .

5.9 Ammonia solution, 25 % solution, $c(\text{NH}_3 \cdot \text{H}_2\text{O}) = 14 \text{ mol/l}$, $\rho(\text{NH}_3 \cdot \text{H}_2\text{O}) = 0,91 \text{ g/ml}$.

5.10 Ethanol, $\text{C}_2\text{H}_5\text{OH}$, a volume fraction of 96 %, $\rho = 0,79 \text{ g/ml}$, food grade or pure grade.

5.11 Sodium sulfate, (Na_2SO_4) , dried for 2 h at 105°C .

5.12 Cation exchange resin, DOWEX 50W-X8¹⁾, particle size 0,3 mm to 0,8 mm in H^+ form.

5.13 Barium chloride solution

In a 1 000 ml volumetric flask, dissolve $917 \text{ mg} \pm 1 \text{ mg}$ of barium chloride dihydrate (5.2) in about 500 ml of water (5.1) and make up to volume with water (5.1).

When stored at 2°C to 6°C , the solution is stable for 1 month.

5.14 Diluted hydrochloric acid, $c(\text{HCl}) = 1 \text{ mol/l}$.

In a 1 000 ml volumetric flask, dilute 82 ml of concentrated hydrochloric acid (5.3) in about 800 ml of water (5.1) and make up to volume with water (5.1).

1) DOWEX 50W-X8 is a trade name. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

5.15 Methylthymol blue solution

In a 1 000 ml volumetric flask, dissolve $160 \text{ mg} \pm 1 \text{ mg}$ of methylthymol blue (5.4) in 50 ml of barium chloride solution (5.13). Add 4 ml of concentrated hydrochloric acid (5.3) and 360 ml of ethanol (5.10). Make up to volume with water (5.1). Add 1 ml of surfactant (5.5).

When stored at 2°C to 6°C , the solution is stable for 1 week.

5.16 Buffered $\text{Na}_4\text{-EDTA}$ solution, $\text{pH} = 10,5$.

In a 1 000 ml volumetric flask, dissolve 6,75 g of ammonium chloride (5.8) and 40,0 g of $\text{Na}_4\text{-EDTA}$ (5.7) in about 500 ml of water (5.1). Add 57 ml of ammonia solution (5.9) and make up to volume with water (5.1).

5.17 Sodium hydroxide solution, $c(\text{NaOH}) = 0,25 \text{ mol/l}$.

In a 1 000 ml volumetric flask, dissolve 10,0 g of sodium hydroxide (5.6) in about 500 ml of water (5.1). Make up to volume with water (5.1).

5.18 Sulfate stock solution, $\rho(\text{SO}_4) = 3\,000 \text{ mg/l}$.

In a 1 000 ml volumetric flask, dissolve $(4\,436 \pm 1) \text{ mg}$ of sodium sulfate (5.11) in about 500 ml of water (5.1). Make up to volume with water (5.1).

5.19 Sulfate spiking solution, $\rho(\text{SO}_4) = 6 \text{ mg/l}$.

In a 1 000 ml volumetric flask, dilute 2,00 ml of sulfate stock solution (5.18) in about 800 ml of water (5.1). Make up to volume with water (5.1). Add 1 ml of surfactant (5.5).

NOTE The sulfate solution is added to achieve better linearity in the lower measuring part of the calibration curve. In respect to the sulfate concentration in the blank or the quality of the methylthymol blue (5.4), the added sulfate amount can be slightly altered. Without the sulfate addition, this lower part will not be linear.

5.20 Calibration solutions

Prepare at least five calibration solutions by diluting the sulfate stock solution (5.18).

Table 1 gives examples for the preparation of 10 calibration solutions in the range of 30 mg/l to 300 mg/l.

Table 1 — Example for preparation of 10 calibration solutions for the sulfate (SO_4) range 30 mg/l to 300 mg/l

Volume in ml of sulfate stock solution (5.18) diluted to 100 ml	1	2	3	4	5	6	7	8	9	10
Sulfate (SO_4) concentration of calibration solution in mg/l	30	60	90	120	150	180	210	240	270	300

6 Apparatus

6.1 Continuous-flow analysis (CFA).

The system generally consists of the following components (see Figure A.1 in Annex A).

6.1.1 Sampler or other device, for reproducible sample introduction.

6.1.2 Reagent containers.