INTERNATIONAL STANDARD

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Water quality — Determination of chromium(VI) — Photometric method for weakly contaminated water

Qualité de l'eau — Dosage du chrome(VI) — Méthode photométrique pour des eaux faiblement contaminées

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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 18412 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Introduction

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

Water quality — Determination of chromium(VI) — Photometric method for weakly contaminated water

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. Care should be taken in handling potassium dichromate due to its carcinogenicity.

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 method for the determination of chromium(VI) in drinking water in mass concentrations between 2 µg/l and 50 µg/l. For the determination of higher concentrations, the sample is diluted prior to analysis. The method may also be applied to weakly polluted ground and surface water, provided the matrix does not contain interfering reducing agents. This method has not been verified for estuarine water and seawater, so the user is responsible for the validation of the method for these matrices. The photometric determination of chromium(VI) in waste water is carried out according to ISO 11083, *Water quality — Determination of chromium(VI) — Spectrometric method using 1,5-diphenylcarbazide*.

2 Normative references

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

ISO 5667-2, *Water quality — Sampling — Part 2: Guidance on sampling techniques*

ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of 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

Reducing agents in the sample may lead to negative bias for the chromium(VI) concentration. Concentrations of sulfide up to 0,2 mg/l do not interfere.

Oxidizing agents for disinfection in drinking water production, such as chlorine, chlorine dioxide, ozone and hydrogen peroxide, do not interfere with the method, provided their mass concentration does not exceed the concentrations given in Table 1.

Table 1 — Mass concentrations of disinfection agents not producing significant interference under the given measuring conditions

Disinfectant	Concentration mg/l
Chlorine	0,6
Chlorine dioxide	0,4
Hydrogen peroxide	0,2
Ozone	0,1

4 Principle

Chromium(VI) oxidizes 1,5-diphenylcarbazide to 1,5-diphenylcarbazone which forms a red-violet complex with chromium. The relationship between the absorbance of the complex and the chromium(VI) concentration is linear. The maximum absorbance is measured at 540 nm.

5 Reagents

Use only reagents of recognized analytical grade.

5.1 Water, complying with grade 1 as defined in ISO 3696, or distilled water or water of equivalent purity.

5.2 Sulfuric acid, ρ (H_2SO_4) ≈ 1,81 g/ml.

5.3 Orthophosphoric acid, ρ (H_3PO_4) ≈ 1,71 g/ml.

5.4 Acid mixture, H_2SO_4/H_3PO_4.

Into a 500 ml volumetric flask introduce about 200 ml of water (5.1). Add 27 ml of sulfuric acid (5.2) and 33 ml of orthophosphoric acid (5.3), mix and dilute to volume with water (5.1).

5.5 Propanone (acetone), C_3H_6O.

5.6 Diphenylcarbazide (DPC reagent), 1 % solution in acetone (5.5).

Dissolve 1 g of 1,5-diphenylcarbazide ($C_{13}H_{14}N_4O$) in 100 ml of acetone (5.5).

Stored in a brown bottle in a refrigerator, + 2°C to + 8°C, the reagent is stable for about 1 week.

5.7 Potassium chromate stock solution, ρ [Cr(VI)] = 1 g/l.

In a 1 000 ml volumetric flask, dissolve 3,735 g K_2CrO_4 in water (5.1) and dilute to volume with water (5.1).

The solution is, when stored at room temperature, stable for at least 1 year.

5.8 Potassium chromate standard solution I, ρ[Cr(VI)] = 100 mg/l.

Pipette 50 ml of potassium chromate stock solution (5.7) into a 500 ml volumetric flask, and dilute to volume with water (5.1).

Prepare the standard solution on the day of use.

5.9 Potassium chromate standard solution II, ρ [Cr(VI)] = 4 mg/l.

Pipette 20 ml of potassium chromate standard solution (I) (5.8) into a 500 ml volumetric flask, and dilute to volume with water (5.1).

Prepare the solution on the day of use.

6 Apparatus

Usual laboratory apparatus and the following.

6.1 Volumetric flasks, of nominal volume 50 ml, 500 ml or 1 000 ml.

6.2 Volumetric pipettes, of nominal volume 25 ml, 40 ml or 50 ml.

6.3 Automatic pipette, nominal value 500 µl.

6.4 Photometer, equipped with cells of optical path length 50 mm, or filter photometer.

7 Sampling and sample pretreatment

Take samples as specified in ISO 5667-1, ISO 5667-2 and ISO 5667-3.

Store the samples in the refrigerator and analyse them within 4 days after sampling.

The sample should not contain more than 50 µg/l of chromium(VI). Dilute the sample appropriately if the concentration is > 50 µg/l.

8 Procedure

8.1 Measurement

Pipette 40 ml of the sample into a 50 ml volumetric flask.

Add 4 ml of acid mixture (5.4), swirl to mix, add 500 µl of DPC reagent (5.6), and mix again.

Dilute to volume with water (5.1) (absorbance A_1, see Clause 9).

Treat a blank for each batch of samples in the same way, by replacing the 40 ml of sample by 40 ml of water (5.1) (absorbance A_2, see Clause 9).

If the absorbance A_2 exceeds 0,005, take the necessary steps to reduce the blank (e.g. by preparing new reagents).

For compensation of a possible absorbance of the sample, prepare an additional sample by pipetting 40 ml of the sample into a 50 ml measuring flask, add 4 ml of the acid mixture, but omit the addition of DPC reagent, swirl and dilute to volume with water (5.1) (absorbance A_3, see Clause 9).

Let the samples stand for 5 min to 20 min and, using the photometer (6.4), measure the absorbance at 540 nm against water (5.1) in the reference cell.

NOTE If other sample volumes are used, the volumes of the reagents need to be adjusted appropriately.