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

Reference number
ISO 18412:2005(E)

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Foreword

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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.

INTERNATIONAL STANDARD **ISO 18412:2005(E)**

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, $\rho\,(H_2SO_4) \approx 1{,}81$ g/ml.

5.3 Orthophosphoric acid, $\rho\,(H_3PO_4) \approx 1{,}71$ 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, $\rho\,[Cr(VI)] = 1$ 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, $\rho[Cr(VI)] = 100$ 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.

8.2 Calibration according to the standard calibration procedure

For a working range of 5 µg/l to 50 µg/l, proceed, for example, as follows:

Into five 50 ml volumetric flasks, pipette between 50 µl and 500 µl (equidistant concentration steps) of the potassium chromate standard solution II (5.9).

Add water (5.1) to complete to about 40 ml.

Add, to each flask, 4 ml of the acid mixture (5.4), and swirl thoroughly to mix. Then add, to each flask, 500 µl of DPC reagent (5.6) and swirl to mix. Dilute to volume with water (5.1).

After 5 min to 20 min, measure the absorbance in a photometer (6.4) at 540 nm against water (5.1) in the reference cell.

From the measurements, evaluate the linear regression line as specified in ISO 8466-1.

Check the calibration graph regularly, especially when new batches of reagents are used.

If a linear regression line is not achieved, it is also possible to evaluate as specified in ISO 8466-2 (non-linear second-order calibration function).

9 Evaluation

Calculate the mass concentration of chromium(VI), expressed in micrograms per litre (µg/l), in the water sample using Equation (1):

$$\rho = \frac{A_1 - A_2 - A_3}{b} \tag{1}$$

where

ρ is the mass concentration, in micrograms per litre (µg/l), of chromium (VI);

A_1 is the absorbance of the sample at 540 nm;

A_2 is the absorbance of the blank at 540 nm;

A_3 is the absorbance of the sample without added DPC at 540 nm;

b is the sensitivity, i.e. the slope of the calibration curve, in litres per microgram (l/µg).

The evaluation refers to a sample volume of 40 ml. Deviating sample volumes or dilutions need appropriate correction.

10 Expression of results

Values are rounded to the appropriate number of significant figures (e.g. 1 µg/l).

EXAMPLE

Chromium(VI) = 5 µg/l;

Chromium(VI) = 12 µg/l.

11 Test report

The test report shall include at least the following information:

a) a reference to this International Standard (ISO 18412);

b) identification of the sample;

c) results according to Clause 10;

d) sample pretreatment, if applicable;

e) any deviation from this procedure and information on all circumstances that may have affected the result.

Annex A
(informative)

Precision data

An interlaboratory trial was carried out in May 2002 with 23 participating laboratories. The results are given in Table A.1.

Table A.1 — Precision data

Sample	l	n	n_{AP} %	x_{true} µg/l	$\bar{x}$ µg/l	η %	s_R µg/l	CV_R %	s_r µg/l	CV_r %
A	19	34	2,9	8,0	8,5	106,5	0,77	9,0	0,47	5,5
B	20	35	5,4	44,0	44,1	100,3	1,43	3,2	0,56	1,3
C	19	34	0	24,0	23,6	98,5	1,51	6,4	1,23	5,2

l	is the number of laboratories
n	is the number of values
n_{AP}	is the percentage of outliers
x_{true}	is the true value (by convention)
$\bar{x}$	is the total mean
η	is the recovery rate
s_r	is the repeatability standard deviation
CV_r	is the repeatability variation coefficient
s_R	is the reproducibility standard deviation
CV_R	is the reproducibility variation coefficient

Matrix:

A	drinking water, spiked with chromium(VI)
B	drinking water, spiked with chromium(VI)
C	raw water, spiked with chromium(VI)

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