147

INTERNATIONAL STANDARD

ISO 9174

First edition
1990-04-15

Water quality — Determination of total chromium — Atomic absorption spectrometric methods

Qualité de l'eau — Dosage du chrome total — Méthodes par spectrométrie d'absorption atomique

Reference number
ISO 9174:1990(E)

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.

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.

International Standard ISO 9174 was prepared by Technical Committee ISO/TC 147, *Water quality*.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Introduction

Chromium may occur in water in the oxydation states III and VI. The two methods described determine total chromium in both oxidation states, either as acid soluble chromium or as soluble chromium, depending on the sample pre-treatment. The method chosen depends on the concentration of chromium in the water to be examined.

Water quality — Determination of total chromium — Atomic absorption spectrometric methods

Section 1: General

1.1 Scope

This International Standard specifies two methods for the determination of total chromium in water by atomic absorption spectrometry. The two methods are covered in separate sections as follows.

Section 2: Method A — Determination of total chromium by flame atomic absorption spectrometry.

Section 3: Method B — Determination of total chromium by electrothermal atomization atomic absorption spectrometry.

Method A is applicable to the analysis of water and waste water when the concentration range is between 0,5 mg/l and 20 mg/l of chromium. When the concentration is below 0,5 mg/l the determination can be carried out after carefully evaporating an acidified sample to small bulk, taking care to avoid the formation of a precipitate.

IMPORTANT — The use of evaporation will increase the effect of interfering substances and therefore for concentrations below 0,1 mg/l method B is given.

For interferences see clause 2.7.

Method B is applicable to the analysis of water and waste water when the concentration range is between 5 µg/l and 100 µg/l of chromium by injecting a sample volume of 20 µg/l. It is applicable to the determination of higher concentrations by using a smaller sample volume.

For interferences see clause 3.7.

1.2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5667-2:1982, *Water quality — Sampling — Part 2: Guidance on sampling techniques.*

ISO 5667-3:1985, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples.*

Section 2: Methode A — Determination of total chromium by flame atomic absorption spectrometry

2.1 Principle

The method is based on the atomic absorption spectrometric measurement of the chromium content of the acidified sample in a nitrous oxide/acetylene flame. Measurement at a wavelength of 357,9 nm. Addition of lanthanum to reduce matrix interferences.

2.2 Reagents

All reagents shall be of recognized analytical grade. Use deionized water or water distilled from an all glass apparatus. The water used for blank determinations and for preparing reagents and standard solutions shall have a chromium content that is negligible compared with the smallest concentrations to be determined in the samples.

2.2.1 Hydrochloric acid, $\rho \approx 1{,}18$ g/ml.

2.2.2 Nitric acid, $\rho \approx 1{,}42$ g/ml.

2.2.3 Nitric acid, $c(HNO_3) = 1{,}5$ mol/l.

Add 100 ml of nitric acid (2.2.2) to 600 ml of water and dilute to 1 000 ml.

2.2.4 Hydrogen peroxide, 30 % (m/m) solution.

2.2.5 Lanthanum chloride, ($LaCl_3$), solution containing 20 g of La per litre.

Dissolve 23,5 g of lanthanum oxide (La_2O_3), in 200 ml of the hydrochloric acid (2.2.1), dilute to 1 000 ml with water and mix.

CAUTION — Appropriate precautions must be observed when preparing this solution because the reaction of La_2O_3 with HCl is strongly exothermic.

2.2.6 Chromium solutions

2.2.6.1 Chromium, stock solution containing 1,000 g of Cr per litre.

Dry a portion of potassium dichromate ($K_2Cr_2O_7$) at 105 °C ± 2 °C for 2 h. Cool and dissolve 2,825 g ± 0,001 g of the dried potassium dichromate in water, add 5 ml of nitric acid (2.2.2) and dilute to 1 000 ml with water in a one-mark volumetric flask.

1 ml of this standard solution contains 1,00 mg of Cr.

Store this solution in either polyethylene or borosilicate glass containers at room temperature. The solution is stable at room temperature for 1 year if it is stored in the dark and has a pH between 1 and 2.

NOTE 1 Chromium stock solutions are commercially available.

2.2.6.2 Chromium, standard solution containing 50 µg of Cr per millilitre.

Pipette 50 ml of the chromium stock solution (2.2.6.1) into a 1 000 ml one-mark volumetric flask. Add 1 ml of nitric acid (2.2.2), make up to the mark with water and mix.

Store this solution in either polyethylene or borosilicate glass containers at room temperature. The solution is stable at room temperature for 1 month if it is stored in the dark.

2.3 Apparatus

Usual laboratory apparatus and

2.3.1 Atomic absorption spectrometer, equipped with a chromium hollow cathode lamp and a nitrous oxide/acetylene burner, and operated according to the manufacturer's instructions.

CAUTION — It is essential that the manufacturer's safety recommendations are strictly observed when using the nitrous oxide/acetylene flame.

2.3.2 Glassware.

Before use carefully soak all glassware for 24 h in the nitric acid (2.2.3) then rinse thoroughly with water.

IMPORTANT — Do not use glassware which has been cleaned with chromic acid.

2.3.3 Membrane filters, of nominal pore diameter 0,45 µm, washed thoroughly with nitric acid (2.2.3) and rinsed with water (see clause 2.2).

2.4 Sampling and preparation of test portions

2.4.1 Collect samples in polyethylene or borosilicate glass containers which have been previously cleaned with nitric acid (2.2.3) and then rinsed with water.