
International Standard



5666/1

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Water quality — Determination of total mercury by flameless atomic absorption spectrometry — Part 1 : Method after digestion with permanganate-peroxodisulfate

mineralization/

Qualité de l'eau — Dosage du mercure total par spectrométrie d'absorption atomique sans flamme — Partie 1 : Méthode après minéralisation au permanganate-peroxodisulfate

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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 developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5666/1 was developed by Technical Committee ISO/TC 147, *Water quality*, and was circulated to the member bodies in December 1981.

It has been approved by the member bodies of the following countries :

Australia	France	Poland
Austria	Germany, F.R.	Romania
Belgium	Hungary	South Africa, Rep. of
Brazil	India	Spain
Canada	Italy	Sweden
Chile	Japan	Switzerland
China	Mexico	United Kingdom
Czechoslovakia	Netherlands	USSR
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Finland	Norway	

No member body expressed disapproval of the document.

Water quality — Determination of total mercury by flameless atomic absorption spectrometry — Part 1 : Method after digestion with permanganate-peroxodisulfate

0 Introduction

This document constitutes the first part of an International Standard specifying methods for the determination of total mercury in water by flameless atomic absorption spectrometry.

Since various types of water may be tested for the presence of mercury, it has been considered preferable to standardize several methods of determination, which, although they are all based on the same instrumental technique (atomic absorption spectrometry), involve, nevertheless, sufficiently important procedural differences so that their respective fields of application differ significantly.

Thus, this part (part 1) specifies a method of determination after digestion with permanganate-peroxodisulfate and is applicable, in particular, to surface waters and domestic and industrial wastewaters.

Part 2 specifies a method of determination after mineralization by means of ultraviolet radiation and is applicable to drinking water and to candidate drinking water.

Part 3, at present under study, will specify a method of determination after digestion with bromine and will apply to soft water and brines to drinking water and to other types of water containing only small amounts of organic matter.

Each of the three parts describes the method in its entirety and can therefore be used independently of the others.

1 Scope and field of application

This part of ISO 5666 specifies a flameless atomic absorption spectrometric method for the determination of total mercury in water, and, in particular, in surface waters and in domestic and industrial wastewaters.

The method permits determination of as little as 0,05 µg of mercury in the test portion used for the analysis under the optimum instrumental conditions (minimum baseline noise of the apparatus, high spectral purity lamp, and reagents with very low mercury contents). For example if using a test portion of 100 ml, the lower limit of determination is 0,5 µg/l.

The method is not applicable if the concentration of organic matter is such that the quantity of potassium permanganate

specified in 7.3.1 is insufficient to produce a persistent intense violet coloration.

In addition, in the presence of iodide ions, the precision of the method may be perceptibly reduced (see clause 9).

2 Reference

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

3 Principle

Digestion of a test portion, using potassium permanganate and potassium peroxodisulfate at 95 °C, in order to convert all the mercury present to mercury(II).

Reduction of the excess of oxidant with hydroxylammonium chloride and reduction of mercury(II) to metallic mercury by tin(II) chloride.

Entrainment of the mercury in a current of gas at ambient temperature and determination of the mercury, as the monatomic vapour, by flameless atomic absorption spectrometry at a wavelength of 253,7 nm.

4 Reagents

During the analysis, use only the water (4.1) and reagents of recognized analytical grade, the mercury contents of which are as low as possible¹⁾.

4.1 Demineralized-distilled water, or water of equivalent purity, free from mercury.

4.2 Sulfuric acid, $\rho_{20} = 1,84$ g/ml.

4.2.1 Sulfuric acid (4.2) diluted 1 + 1.

CAUTION — Add the acid to the water slowly and with constant stirring to avoid spattering of concentrated acid.

4.3 Nitric acid, $\rho_{20} = 1,42$ g/ml.

1) If the reagents used lead to high results in blank tests, it is necessary to use products of better quality.