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Water quality — Determination of mercury — Methods involving enrichment by amalgamation

*Qualité de l'eau — Dosage du mercure — Méthodes après enrichissement
par amalgame*



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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 16590 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Annexes A, B and C of this International Standard are for information only.

This International Standard is equivalent to EN 12338.

Water quality — Determination of mercury — Methods involving enrichment by amalgamation

WARNING — Mercury and mercury compounds are very toxic. Extreme caution is recommended when handling samples and solutions which contain or may contain mercury.

1 Scope

This International Standard specifies two methods for the determination of mercury, one using tin(II) chloride and the other sodium tetrahydroborate as reducing agent. The methods are suitable for the determination of mercury in water, for example in ground, surface or waste water, in the concentration range 0,01 µg/l to 1 µg/l. Higher concentrations may be determined if the water sample is diluted.

The total mercury content of the water is determined after digestion of the sample. If only soluble mercury compounds are to be determined, the sample is filtered through a 0,45 µm membrane filter prior to digestion.

Mono- or divalent mercury is reduced to the elemental form by a reducing agent such as tin(II) chloride, SnCl₂, or sodium tetrahydroborate, NaBH₄, in an acid medium. Elemental mercury is then stripped from the solution with the aid of a stream of inert gas with negligible mercury content and transported over a noble-metal surface with a large area, such as gold/platinum gauze, on which the mercury is adsorbed. The mercury is released by rapid heating of the adsorbent and further transported in a stream of carrier gas to a suitable cuvette. Absorbances are measured at a wavelength of 253,7 nm in the radiation beam of an atomic absorption spectrometer. Concentrations are calculated using a calibration curve.

In order to fully decompose all of the mercury compounds, a digestion procedure is required. Digestion can be omitted only if it is certain that the mercury concentration may be measured without this pretreatment.

Careful consideration should be given to whether, and to what extent, particular problems will require the specification of additional conditions.

It is absolutely essential that analyses conducted in accordance with this International Standard are carried out by suitably qualified staff.

In natural water sources, mercury compounds generally occur only in very small concentrations of less than 0,1 µg/l. Higher concentrations may be found, for example, in waste water. Both inorganic and organic compounds of mercury can be present. Mercury may accumulate in sediment and sludge.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 5667-1:1980, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes*.

ISO 5667-2:1991, *Water quality — Sampling — Part 2: Guidance on sampling techniques*.

ISO 5667-3:1994, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples.*

3 General interferences

With mercury, there is a risk that exchange reactions, that is adsorption and desorption, will occur on the walls of the reaction vessel. It is therefore essential that 4.6 is followed exactly.

Mercury vapour can diffuse through various plastics. Therefore, special consideration shall be given to the choice of tubing material. Glass or special plastics tubing, e.g. FEP tubes, may be used (FEP = perfluoroethene-hexafluoropropene copolymer). Silicone tubing, for example, is unsuitable.

Volatile organic substances can absorb in the UV range and be mistaken for mercury. They are in most cases destroyed by adding potassium permanganate until the solution is permanently coloured red and removed by purging for 10 min with an inert gas, prior to reduction of the mercury compounds. Often, such interference by non-specific absorption can also be eliminated by using a background compensation system.

All solutions shall be brought to the same temperature (<25 °C) before reduction and stripping of the mercury vapour. Water condensation on the cuvette windows can be prevented by heating the cuvette with, for example, an infrared lamp.

The interference which occurs due to the presence of other elements in the matrix depends on the choice of reducing agent. Element concentrations in excess of those listed in Table 1 may cause too low results.

Less interference arises from heavy metals if tin(II) chloride is used rather than sodium tetrahydroborate. When flow systems are used, interference effects due to heavy metals may be less than indicated in Table 1.

Tin(II) chloride causes such extensive contamination of the apparatus with tin that considerable interference occurs if sodium tetrahydroborate is used afterwards. Separate systems are therefore used for reductions with tin(II) chloride and with sodium tetrahydroborate.

Table 1 — Acceptable concentrations of some matrix elements in the test solution

Reducing agent	NaBH ₄	NaBH ₄	SnCl ₂
Medium	0,5 mol/l HCl	5 mol/l HCl + 0,2 g/l Fe(III)	0,5 mol/l HCl
Element	Acceptable concentration (mg/l)		
Cu(II)	10	10	500
Ni(II)	1	500	500
Ag(I)	0,1	10	1
I ⁻	100	10	0,1
As(V)	0,5	0,5	0,5
Bi(III)	0,05	0,5	0,5
Sb(III)	0,5	0,5	0,5
Se(IV)	0,005	0,05	0,05