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INTERNATIONAL STANDARD

NORME INTERNATIONALE

HORIZONTAL STANDARD

NORME HORIZONTALE

Determination of certain substances in electrotechnical products – Part 4: Mercury in polymers, metals and electronics by CV-AAS, CV-AFS, ICP-OES and ICP-MS

Détermination de certaines substances dans les produits électrotechniques – Partie 4: Mercure dans les polymères, métaux et produits électroniques par CV-AAS, CV-AFS, ICP-OES et ICP-MS

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FOREWORD

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International Standard IEC 62321-4 has been prepared by IEC technical committee 111: Environmental standardization for electrical and electronic products and systems.

It has the status of a horizontal standard in accordance with IEC Guide 108.

The first edition of IEC 62321:2008 was a 'stand alone' standard that included an Introduction, an overview of test methods, a mechanical sample preparation as well as various test method clauses.

This first edition of IEC 62321-4 is a partial replacement of IEC 62321, forming a structural revision and replacing Clause 7 and Annex E.

Future parts in the IEC 62321 series will gradually replace the corresponding clauses in IEC 62321:2008. Until such time as all parts are published, however, IEC 62321:2008 remains valid for those clauses not yet re-published as a separate part.

The text of this standard is based on the following documents:

FDIS	Report on voting
111/299/FDIS	111/309/RVD

Full information on the voting for the approval of this standard can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts in the IEC 62321 series can be found on the IEC website under the general title: *Determination of certain substances in electrotechnical products*

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC web site under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

INTRODUCTION

The widespread use of electrotechnical products has drawn increased attention to their impact on the environment. In many countries this has resulted in the adaptation of regulations affecting wastes, substances and energy use of electrotechnical products.

The use of certain substances (e.g. lead (Pb), cadmium (Cd) and polybrominated diphenyl ethers (PBDEs)) in electrotechnical products, is a source of concern in current and proposed regional legislation.

The purpose of the IEC 62321 series is therefore to provide test methods that will allow the electrotechnical industry to determine the levels of certain substances of concern in electrotechnical products on a consistent global basis.

WARNING – Persons using this International Standard should be familiar with normal laboratory practice. This 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.

DETERMINATION OF CERTAIN SUBSTANCES IN ELECTROTECHNICAL PRODUCTS –

Part 4: Mercury in polymers, metals and electronics by CV-AAS, CV-AFS, ICP-OES and ICP-MS

1 Scope

This part of IEC 62321 describes test methods for mercury in polymers, metals and electronics by CV-AAS, CV-AFS, ICP-OES and ICP-MS.

This standard specifies the determination of the levels of mercury (Hg) contained in electrotechnical products. These materials are polymers, metals and electronics (e.g. printed wiring boards, cold cathode fluorescent lamps, mercury switches). Batteries containing Hg should be handled as described in [1][1]. The interlaboratory study has only evaluated these test methods for plastics, other matrices were not covered.

This standard refers to the sample as the object to be processed and measured. What the sample is or how to get to the sample is defined by the entity carrying out the tests. Further guidance on obtaining representative samples from finished electronic products to be tested for levels of regulated substances may be found in IEC 62321-2. It is noted that the selection and/or determination of the sample may affect the interpretation of the test results.

This standard describes the use of four methods, namely CV-AAS (cold vapour atomic absorption spectrometry), CV-AFS (cold vapour atomic fluorescence spectrometry) ICP-OES (inductively coupled plasma optical emission spectrometry), and ICP-MS (inductively coupled plasma mass spectrometry) as well as several procedures for preparing the sample solution from which the most appropriate method of analysis can be selected by experts.

Analysis by CV-AAS, CV-AFS, ICP-OES and ICP-MS allows the determination of the target element, mercury, with high precision (uncertainty in the low per cent range) and/or high sensitivity (down to the μg/kg level). The test procedures described in this standard are intended to provide the highest level of accuracy and precision for concentrations of mercury in the range from 4 mg/kg to 1 000 mg/kg. The procedures are not limited for higher concentrations.

For direct analysis, using thermal decomposition-gold amalgamation in conjunction with CV-AAS (TD(G)-AAS) can be also applied for mercury analysis without sample digestion, although the detection limits are higher than other methods due to the reduced sample size.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 62321-1, *Determination of levels of certain substances in electrotechnical products – Part 1: Introduction and overview*

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1 Figures in square brackets refer to the bibliography.

IEC 62321-2, *Determination of levels of certain substances in electrotechnical products – Part 2: Disassembly, disjointment and mechanical sample preparation*[2]

IEC 62321-3-1, *Determination of certain substances in electrotechnical products – Part 3-1: Screening – Lead, mercury, cadmium, total chromium and total bromine by X-ray fluorescence spectrometry*

IEC 62554, S*ample preparation for measurement of mercury level in fluorescent lamps*

ISO 3696, *Water for analytical laboratory use – Specification and test methods*

3 Terms, definitions and abbreviations

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC 62321-1 as well as the following, apply.

3.1.1
accuracy
closeness of agreement between a test result and an accepted reference value

3.1.2
blank calibration solution
calibration solution without analyte

3.1.3
calibration standard
substance in solid or liquid form with known and stable concentration(s) of the analyte(s) of interest used to establish instrument response (calibration curve) with respect to analyte(s) concentration(s)

3.1.4
calibration solution
solution used to calibrate the instrument prepared either from (a) stock solution(s) or from a (certified) reference material

3.1.5
certified reference material
reference material, accompanied by documentation issued by an authoritative body and providing one or more specified property values with associated uncertainties and traceabilities using valid precedures

3.1.6
laboratory control sample
known matrix spiked with compound(s) representative of the target analytes, used to document laboratory performance

[SOURCE: US EPA SW-846] [2]

3.1.7
reagent blank solution
prepared by adding to the solvent the same amounts of reagents as those added to the test sample solution (same final volume)

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2 To be published.

3.1.8
stock solution
solution with accurately known analyte concentartion(s), prepared from “pure chemicals”

3.1.9
test portion
quantity of material drawn from the test sample (or from the laboratory sample if both are the same) and on which the test or observation is actually carried out

[SOURCE ISO 6206:1979] [3]

3.1.10
test sample solution
solution prepared with the test portion of the test sample according to the appropriate specifications such that it can be used for the envisaged measurement

3.2 Abbreviations

CRM	Certified reference material
CCFL	Cold cathode fluorescent lamp
CCV	Continuing calibration verification
CV-AAS	Cold vapour atomic absorption spectrometry
CV-AFS	Cold vapour atomic fluorescence spectrometry
LCS	Laboratory control sample
LOD	Limits of detection
LOQ	Limits of quantification
MDL	Method detection limit
TD(G)-AAS	Thermal decomposition – Gold amalgamation – Atomic absorption spectrometry

NOTE TD(G)-AAS is commonly referred to as a direct mercury analysis or DMA technique.

4 Reagent and materials

4.1 General

For the determination of elements at trace level, the reagents shall be of adequate purity. Contamination can be a major source of error when working in the 1 ng range with the instruments. Cautious handling of the apparatus and careful technique will minimize this problem. Therefore, only grade 1 water (4.2 a) shall be used. Care shall be taken that all materials in contact with the water are Hg-free.

Chemicals used for sample preparation can be a major source of contamination. Only reagents that are mercury-free shall be used. It is therefore highly recommended that the blank values of the reducing agents and the other chemicals be measured before using them for sample preparation.

4.2 Reagents

The following reagents are used:

a) Water: Grade 1, as defined in ISO 3696, shall be used for preparation and dilution of all sample solutions.

b) Nitric acid (concentrated nitric acid): $\rho(HNO_3)$ = 1,4 g/ml , a mass fraction of 65 %, trace metal grade.

c) Nitric acid, a mass fraction of 50 %, trace metal grade.

d) Nitric acid, 0,5 mol/l, trace metal grade.

e) Nitric acid, a mass fraction of 1 %, trace metal grade.

f) Nitric acid, a mass fraction of 1,5 %, trace metal grade.

g) Nitric acid, a mass fraction of 5 % , trace metal grade.

h) Fluoroboric acid: HBF_4, a mass fraction of 50 %, trace metal grade (for microwave digestion).

i) Hydrogen peroxide: H_2O_2, a mass fraction of 30 %, trace metal grade (for microwave digestion).

j) Stock solution with 1 000 mg/L of mercury, trace metal grade.

k) Potassium tetrahydridoborate (potassium borohyride): KBH_4, trace metal grade.

l) Potassium permanganate: $KMnO_4$, a mass fraction of 5 % solution, trace metal grade. Dissolve 5 g of potassium permanganate in 100 ml of water (4.2 a).

m) Sodium tetrahydridoborate (sodium borohydride), $NaBH_4$, trace metal grade.

n) Sodium hydroxide, NaOH trace metal grade.

o) Hydrogen tetrachloroaurate (Ⅲ) tetra hydrate, $HAuCl_4$ □4H2O trace metal grade.

p) Internal standard stock solution, trace metal grade:

- Internal standard elements that do not interfere with the target element are used for ICP-OES and ICP-MS. Also, the presence of these internal standard elements in the sample solution shall be at negligible levels. Sc, In, Tb, Lu, Re, Rh, Bi and Y may be used as internal standard elements.
- For use with ICP-OES, Sc or Y is recommended. The recommended concentration is 1 000 mg/L.
- For use with ICP-MS, Rh is recommended. The recommended concentration is 1 000 μg/l.

q) Reducing agent for CV-AAS: a mass fraction of 3 % $NaBH_4$ in a mass fraction of 1 % NaOH.

Dissolve 10,0 g sodium hydroxide (4.2 n) into approximately 700 ml of water (4.2 a) in a beaker and stir until dissolved. Add 30,0 g of sodium tetrahydridoborate powder (4.2 m) into the beaker and stir until dissolved. Finally transfer to a 1 l volumetric flask and fill up to the mark with water (4.2 a) and filter. Prepare daily.

Reductant solution containing sodium tetrahydridoborate in a sodium hydroxide solution is recommended. If the available mercury hydride system is incompatible with this reductant, tin (II) chloride or stannous sulfate can be used instead. The instructions given in the operator's manual for the instrument should be followed.

r) Reducing agent for CV-AFS: a mass fraction of 1 % (m/v) KBH_4 in a mass fraction of 0,05 % NaOH.

Dissolve 0,50 g sodium hydroxide (4.2 n) into approximately 700 ml of water (4.2 a) in a beaker and stir until dissolved. Add 10,0 g of potassium tetrahydridoborate (4.2 k) into the beaker and stir until dissolved. Finally transfer to a 1 l volumetric flask and fill up to the mark with water (4.2 a) and filter. Prepare daily.

Reductant solution containing potassium tetrahydridoborate in a sodium hydroxide solution is recommended. If the available mercury hydride system is incompatible with this reductant, tin (II) chloride or stannous sulfate can be used instead. The instructions given in the operator's manual for the instrument should be followed.

s) Gold preservation stock solution for mercury (1 ml = 100 μg): it is recommended purchasing as high purity prepared solution of $AuCl_3$ in dilute hydrochloric acid matrix.

t) Diatomaceous earth

Analytical grade reagents may be used as an alternative except when utilizing ICP-MS methods.

4.3 Materials

Materials include:

a) Mercury collector for thermal–decomposition-gold amalgamation system

A solution of 1 g of hydrogen tetrachloroaurate(Ⅲ) tetra hydrate (4.2 o) in 20 ml to 30 ml of water (4.2 a) is added to 3 g of 420 μm to 590 μm diatomaceous earth, which is then mixed until homogeneous. After being dried at approximately 80 °C, the collector is loaded into a tube furnace and heated for 30 min at around 800 °C in flowing air.

5 Apparatus

5.1 General

In general, the collection and storage of glassware are a critical part of mercury analysis, regardless of the type of sample to be analysed. Because of the sensitivity of the mercury analysis techniques described, each individual sampling step shall be carried out with great care.

Beakers, pipettes, volumetric flasks, etc. are all major sources of metal contamination. It is essential to use mercury-free plastic or quartz glassware for sample handling.

All sampling, storage and manipulation apparatus shall be mercury free. Soak all glassware in 50 % nitric acid (4.2 c) for 24 h at room temperature, and then rinse thoroughly with water (4.2 a).

For measurements by ICP-OES and ICP-MS, the memory effect occurs in cases where high concentrations of mercury are introduced. Dilution of the sample solution is required for high levels of mercury. If the memory effect is not decreased by dilution, thorough washing of the equipment is required.

5.2 Apparatus

The following apparatus shall be used:

a) Analytical balance capable of measuring accurately to 0,000 1 g.

For wet digestion as described in 7.1:

b) Heating digester equipped with reaction vessels, reflux coolers and absorption vessels (for the digestion of metals and electronics).

c) Glass fibre filter 0,45 μm.

For microwave digestion as described in 7.2:

d) Microwave sample preparation system equipped with a sample holder and high-pressure polytetrafluoroethylene/tetrafluoroethylene modified (PTFE/TFM) or perfluoro alkoxyl alkane resin /tetrafluoroethylene modified (PFA/TFM) or other vessels based on fluorocarbon materials (for the digestion of metals containing significant amounts of silicon (Si), zirconium (Zr), hafnium (Hf), titanium (Ti), tantalum (Ta), niobium (Nb) or tungsten (W), and for plastics).

e) Glass microfibre filter (borosilicate glass), pore size: 0,45 μm and a suitable filter cup.

f) Volumetric flasks such as 25 ml, 250 ml , etc. (PTFE-PFA equipment or glassware). Where appropriate, other types of volumetric equipment with acceptable precision and accuracy can be used as alternatives to volumetric flasks.

g) Pipettes such as 1 ml, 2 ml, 5 ml, 10 ml, etc. (PTFE-PFA equipment or glassware).

h) Micropipettes such as 200 μl, 500 μl, 1 000 μl, etc.

i) Plastic containers for standards and digestion solutions. (PTFE-PFA equipment).

j) Cold vapour atomic absorption spectrometer (CV-AAS).

k) Cold vapour atomic fluorescence spectrometer (CV-AFS).

l) Inductively coupled plasma optical emission spectrometer (ICP-OES).

m) Inductively coupled plasma mass spectrometer (ICP-MS).

n) Argon gas with a purity of at least 99,99 %.

o) Thermal decomposition-gold amalgamation system.

6 Sampling and test portion

The different test methods, which can be used as alternatives according to this standard, need different amounts of sample to obtain the required quality of results.

In the case of electronics, the sample shall first be destroyed mechanically by appropriate means (e.g. grinding, milling, mill cutting with LN_2-cooling due to volatility of mercury) before chemical dissolution of the powder can start. To ensure representative sample taking at this stage, a certain particle size as a function of the starting amount of sample is required (see IEC 62321-2).

For the determination of mercury in fluorescent self ballasted lamps, single capped compact florescent multi lamps and linear fluorescent lamps, follow the instructions given in IEC 62554.

If using a thermal decomposition-gold amalgamation system, samples should be milled in a ball mill and homogenized in advance. Difficult samples, like metals, to be ground as finely as possible. Put 50 mg to 200 mg of the sample into a sample boat. If using an additive, spread 0,5 g in a thin layer over the surface of the sample boat, evenly spread the sample over the additive, and then cover the sample with 2 g of additive.

It is recommended to analyse aqueous sample solutions containing mercury preferably directly after sample preparation. If this is not possible, it is highly recommended stabilizing the solutions in an adequate way, and to store the solutions no longer than 28 days at ambient temperature.

7 Procedure

7.1 Wet digestion (digestion of electronics)

Wet digestion is recommended for the digestion of metals and electronics, with the exception of metals containing significant amounts of Si, Zr, Hf, Ti, Ta, Nb or W. For these materials and for polymers, microwave digestion, as described in 7.2, is recommended.

a) Weigh 1 g of a sample to the nearest 0,1 mg into the reaction vessel and 30 ml concentrated nitric acid (4.2 b) is added. (When the available sample amount is 500 mg or less, refer to the instructions given in 7.2 a).

   The vessel is equipped with a reflux cooler and an absorption vessel (on top of the reflux cooler – see Figure A.1) containing 10 ml 0,5 mol/l nitric acid (4.2 d). A temperature program is then started to digest the samples for 1 h at room temperature and for 2 h at 90 °C.

   After cooling to room temperature, the contents of the absorption tube are placed in the reaction vessel and the solution obtained is transferred to a 250 ml volumetric flask (5.2 f) and filled with 5 % nitric acid (4.2 g) to the mark (if the sample is digested completely).

b) For ICP-OES and ICP-MS measurements, the sample solution obtained may be diluted with water (4.2 a) to the appropriate concentration levels for measurements. Add 250 μl of internal standard (4.2 p) for a volume of 250 ml before filling to the mark.

c) If the sample is not completely digested (e.g. printed wiring boards), the sample is filtered with a filter (5.2 e) and the solid residue is washed four times with 15 ml 5 % nitric acid

(4.2 g). The solution obtained is transferred to a 250 ml volumetric flask (5.2 f) and filled with 5 % nitric acid (4.2 g) to the mark.

d) Any sample residues shall be separated by a centrifuge or a filter. The residues shall be tested by appropriate measurements (e.g. XRF, alkali fusion method, other acid digestion methods, etc.) to confirm the absence of target elements. The instruction for XRF is given in IEC 62321-3-1.

7.2 Microwave digestion

Microwave digestion is recommended for the following materials:

– metals containing significant amounts of Si, Zr, Hf, Ti, Ta, Nb or W,

– polymers,

in cases where the available sample amount is smaller than 500 mg.

It is highly recommended that the same sample amounts and the same type of samples be weighed in one digestion run.

NOTE 1 Mercury can be determined in the same solution with Pb and Cd obtained in a closed system for acid decomposition, as described in IEC 62321-5 [4].

a) Weigh, 0,1 g of a sample to the nearest 0,1 mg into a PTFE-TFM or PFA-TFM vessel. Add 5 ml of concentrated nitric acid (4.2 b), 1,5 ml 50 % HBF_4 solution (4.2 h), 1,5 ml 30 % H_2O_2 (4.2 i) and 1 ml water (4.2 a). Close the vessel and digest the sample in the microwave oven following a digestion program specified in advance. An example of a suitable microwave program is given in Annex A.

NOTE 2 If HBF_4 is not available in sufficient purity, HF may be used as an alternative.

Hydrogen peroxide should only be added when the reactive components of the sample are known. Hydrogen peroxide may react rapidly and violently with easily oxidizable materials and should not be added if the sample contains large quantities of easily oxidizable organic constituents.

b) Cool the vessel to room temperature (approximately 1 h). Open the vessel, filter the solution with filter (5.2 e) into a 25 ml flask (5.2 f), wash with water (4.2 a) and fill to mark with water (4.2 a).

c) Any sample residues shall be separated by a centrifuge or filter. The residues shall be checked by appropriate measurements (e.g. XRF, alkali fusion method, other acid digestion methods, etc.) to confirm the absence of target elements. The instruction for XRF is given in IEC 62321-3-1.

The resulting concentrated solutions may be measured directly by ICP-OES and ICP-MS, i.e. the digestion solution may be analysed without any further sample preparation. When using CV-AAS and CV-AFS, the mercury is reduced to its elemental state before it is analysed.

7.3 Thermal decomposition-gold amalgamation system

The procedure should be performed as follows, but also follow the instruction manual of the relevant instruments for details on their operation:

a) Place the sample vessel charged with a sample in position in the automatic sample changer.

b) Set the predetermined temperature ramp program and raise the temperature of the sample heating furnace.

c) The mercury, mercury compounds and combustion product gases generated from the sample will be decomposed in the decomposition furnace containing the catalyst and then scrubbed and dehumidified in the gas washing bottle and the dehumidifier bottle.