BS EN 62321-4:2014

BSI Standards Publication

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

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National foreword

This British Standard is the UK implementation of EN 62321-4:2014. It is identical to IEC 62321-4:2013. Together with BS EN 62321-1:2013, BS EN 62321-2:2014, BS EN 62321-3-1:2014, BS EN 62321-3-2:2014, BS EN 62321-5:2014, BS EN 62321-6, BS EN 62321-7-1, BS EN 62321-7-2 and BS EN 62321-8 it supersedes BS EN 62321:2009, which will be withdrawn upon publication of all parts of the BS EN 62321 series.

The UK participation in its preparation was entrusted to Technical Committee GEL/111, Electrotechnical environment committee.

A list of organizations represented on this committee can be obtained on request to its secretary.

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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
(IEC 62321-4:2013)

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
(CEI 62321-4:2013)

Verfahren zur Bestimmung von bestimmten Substanzen in Produkten der Elektrotechnik - Teil 4: Quecksilber in Polymeren, Metallen und Elektronik mit CV-AAS, CV-AFS, ICP-OES und ICP-MS
(IEC 62321-4:2013)

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European Committee for Electrotechnical Standardization
Comité Européen de Normalisation Electrotechnique
Europäisches Komitee für Elektrotechnische Normung

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Foreword

The text of document 111/299/FDIS, future edition 1 of IEC 62321-4, prepared by IEC/TC 111 "Environmental standardization for electrical and electronic products and systems" was submitted to the IEC-CENELEC parallel vote and approved by CENELEC as EN 62321-4:2014.

The following dates are fixed:

• latest date by which the document has to be implemented at national level by publication of an identical national standard or by endorsement	(dop)	2014-10-25
• latest date by which the national standards conflicting with the document have to be withdrawn	(dow)	2016-11-15

EN 62321-4:2014 is a partial replacement of EN 62321:2009, forming a structural revision and replacing Clause 7 and Annex E.

Future parts in the EN 62321 series will gradually replace the corresponding clauses in EN 62321:2009. Until such time as all parts are published, however, EN 62321:2009 remains valid for those clauses not yet re-published as a separate part.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CENELEC [and/or CEN] shall not be held responsible for identifying any or all such patent rights.

Endorsement notice

The text of the International Standard IEC 62321-4:2013 was approved by CENELEC as a European Standard without any modification.

In the official version, for Bibliography, the following note has to be added for the standard indicated:

IEC 62321-5 NOTE Harmonised as EN 62321-5.

Annex ZA
(normative)

Normative references to international publications with their corresponding European publications

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.

NOTE When an international publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

Publication	Year	Title	EN/HD	Year
IEC 62321-1	-	Determination of certain substances in electrotechnical products - Part 1: Introduction and overview	EN 62321-1	-
IEC 62321-2	-	Determination of certain substances in electrotechnical products - Part 2: Disassembly, disjunction and mechanical sample preparation	EN 62321-2	-
IEC 62321-3-1	-	Determination of certain substances in electrotechnical products - Part 3-1: Screening electrotechnical products for lead, mercury, cadmium, total chromium and total bromine using X-ray Fluorescence Spectrometry	EN 62321-3-1	-
IEC 62554	-	Sample preparation for measurement of mercury level in fluorescent lamps	EN 62554	-
ISO 3696	-	Water for analytical laboratory use - Specification and test methods	EN ISO 3696	-

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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.

d) The mercury and other gases are introduced into the mercury collecting tube, where only mercury is trapped in the form of amalgam, and any other gases discharged through the switching valve.

e) The mercury collecting tube is heated at a constant temperature of 350 °C to 600 °C, and the generated mercury introduced into the absorption cell or the fluorescence cell. The height or area of the absorption peak or the fluorescence intensity is then measured at a wavelength of 253,7 nm.

7.4 Preparation of reagent blank solution

The procedure is identical to that of sample preparation and is carried out concurrently but without the sample.

8 Calibration

8.1 General

All analyses require that a calibration curve shall be prepared to cover the appropriate concentration range. Calibration solutions are prepared by diluting the stock solution (4.2 j) with 1,5 % nitric acid (4.2 f). When internal standard methods (ICP-OES and ICP-MS) are used, the appropriate amounts of solution for the internal standard stock solutions (4.2 p) are added.

Prepare a reagent blank solution of 1,5 % nitric acid (4.2 f) and at least three calibration solutions in graduated amounts in the appropriate range of the linear part of the calibration curve.

Calibration solutions shall be stored in mercury-free plastic containers. The stock solution (4.2 j) is usually stable for at least a year, whereas calibration solutions shall be prepared daily.

The stability of mercury calibration solutions can be severely affected by adsorption on the walls of the storage vessel. Therefore, it is recommended that mercury calibration solutions be stabilized by the addition of a few drops of 5 % $KMnO_4$ (4.2 l) solution.

NOTE A 1 % gold (Au) solution can also be used instead of potassium permanganate.

8.2 Development of the calibration curve

The spectrometers are prepared for quantification with a reagent blank solution and a minimum of three calibration solutions.

a) CV-AAS
   1) The readings for the absorbance of the target element mercury are determined. The calibration curve obtained shows the relationship between the absorbance of mercury and its concentration.
   2) The recommended wavelength and examples of workable instrument parameters are listed in Clause A.3.

b) CV-AFS
   1) The readings for the fluorescence intensity of the target element mercury are determined. The calibration curve obtained shows the relationship between the fluorescence intensity of mercury and its concentration.
   2) The recommended wavelength and examples of workable instrument parameters are listed in Clause A.3.

c) ICP-OES

   The readings for the emission intensity of the target element mercury and those of the internal standard are determined. The calibration curve obtained shows the relationship between the ratio of emission intensities of mercury and those of the internal standard to the concentration of mercury.

The recommended wavelength for mercury and examples of workable instrument parameters are listed in Clause A.3.

d) ICP-MS

The readings for the mass/charge (m/z) intensity of the target element mercury and those of the internal standard are determined. The calibration curve obtained shows the relationship between the intensity ratio of the m/z of mercury and that of the internal standard to the concentration of mercury.

The recommended m/z ratios for mercury and examples of workable instrument parameters are listed in Clause A.3.

e) TD(G)-AAS

Four or five calibration solutions, including a blank calibration solution, are placed directly into the sample boats using a micro pipette while changing the amount but ensuring it is within the working measurement range, and measured in the same manner as samples. A calibration curve is then derived from the relationship between the amounts of mercury and indicated values. For example, in the case of a sample's mercury concentration being around 10 mg/kg, use 50 µl, 100 µl, 150 µl and 200 µl of 100 µg/ml stock solution for measurement and develop a calibration curve from the results obtained.

8.3 Measurement of the sample

After development of the calibration curve, the reagent blank solution and the test sample solutions are measured. If the sample concentration is above the range of the concentration curve, the solution shall be diluted with 1 % nitric acid (4.2 e) to the range of the calibration curve and measured again.

Measurement precision and baseline drift shall be checked with a standard substance, calibration solution, etc. and a blank calibration solution at regular intervals (such as every 10 samples) and after the last sample.

If the sample is diluted to the range of calibration, it should be ensured that the internal standard concentration in the diluted sample solution is adjusted to the standard solution.

9 Calculation

The concentration measured in 8.3 is the concentration of mercury in the sample solution. The concentration of mercury in the sample is calculated from the following formula:

$$c = \frac{(A_1 - A_2)}{m} \times V \tag{2}$$

where

c is the concentration of mercury in the sample in µg/g;

A_1 is the concentration of mercury in the sample solution in mg/l;

A_2 is the concentration of mercury in the reagent blank solution in mg/l;

V is the total volume for the sample solution in ml which depends on

- the type of digestion carried out (250 ml for wet digestion, 25 ml for microwave digestion),
- the type of the particular series of dilutions used;

m is the measured quantity of the sample in g.

10 Precision

When the values of two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, lie within the range of the mean values cited in Table 1 below, the absolute difference between the two test results obtained will not exceed the repeatability limit, *r*, deduced by statistical analysis on the international interlaboratory study Nos. 2 (IIS2) and 4A (IIS 4A) results in more than 5 % of cases (see Annex B).

When the values of two single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, lie within the range of the values cited Table 1 below, the absolute difference between the two results will not be greater than the reproducibility limit *R* by statistical analysis on interlaboratory study Nos. 2 (IIS2) and 4A (IIS 4A) results in more than 5 % of cases.

Table 1 – Repeatability and reproducibility

Material type	**IIS**	**Technique**	**Mean value** mg/kg	***r*** mg/kg	***R*** mg/kg
Polymer	2	CV-AAS	21,4	7,0	Insufficient data
			4,2	0,4	
			88,5	3,3	13,7
			883,5	45,1	165,1
	4A		30,0	1,6	3,7
			52,6	4,1	10,6
	2	CV-AFS	24,8	0,6	Insufficient data
			4, 8	0,4	
	4A		30, 0	5,3	15,0
			52,6	7,1	27,2
	4A	TD(G)-AAS	31,3	0,7	4,2
			56,8	2,1	8,7
			22,7	2,4	5,5
	2	ICP-OES	25,4	7,2	19,2
			3,8	1,4	Insufficient data
			90,7	8,8	23,1
			901,7	116,2	192,7

NOTE Repeatability and reproducibility data for the ICP-MS technique as well as metal electronic material types are not available due to limited availability of participating laboratories and and appropriate samples for international interlaboratoty study. Results similar to those shown above are anticipated for the ICP-MS technique as well as metal electronic material types

See Annex B for supporting data.

11 Quality assurance and control

11.1 General

Where applicable, the quality assurance and control clauses of the individual test method standards shall include control sample requirements regarding testing frequency and acceptance criteria. This clause shall also include method specific quality control concerns regarding the determination of limits of detection (LOD) and limits of quantification (LOQ). Where applicable, the LOD and LOQ section shall be consistent with the descriptions in 11.2.

Examples of other method specific quality control concerns include requirements regarding initial calibration verification, method blanks, laboratory control samples (LCS) and so forth are listed in Table 2.

Table 2 – Acceptance criteria of items for the quality control

Items	**Concentration** mg/kg in test sample	**Acceptance criteria**
Calibration curve		$R^2 > 0,995$
Initial calibration verification	e.g.) 1 mg/kg for Hg	Recovery: (90 to 110) %
Continuing calibration verification (CCV)	e.g.) 1 mg/kg for Hg	Recovery: (90 to 110) %
Method blank		< MDL
Laboratory control sample (LCS)	Middle of calibration range	Recovery: (80 to 120) %
Laboratory control sample duplicate	Middle of calibration range	Relative deviation <20 %

a) Initial calibration verification is performed whenever a calibration curve is established, using a standard from a source different than calibration standard.

b) One method blank should be analysed at once per a batch. A blank matrix which does not contain mercury can be used as a method blank sample.

c) One laboratory control sample (LCS) and laboratory sample duplicate per batch, should be analysed by means of spiking mercury in the blank matrix. Alternatively, a certified reference material containing mercury can be tested in duplicate.

d) After every tenth sample run and at the end of each sample set, analyse a continuing calibration verification standard (CCV). The percent recovery for mercury shall be between 90 % and 110 %. If the per cent recovery for mercury in the CCV standard falls outside of this range, the CCV standard should be re-analysed within 12 h. If the recovery is still out of range after re-analysis of the CCV standard, the analysis is stopped and maintenance shall be performed on the system to return it to optimal operating conditions. All samples loaded before the last successful CCV standard may be reported, but all samples after the failing CCV standard shall be re-analysed with a new calibration.

11.2 Limits of detection (LOD) and limits of quantification (LOQ)

The following experimental procedure is performed to determine the method detection limit and limit of quantification for mercury.

a) Accurately weigh out the appropriate amount of sample known not to contain mercury (e.g. certified reference materials) or other compounds that may interfere with the analysis according to the procedure of interest in Clause 7. Place the sample into each vessel (5.2 b or 5.2 d or 5.2 o). Repeat this step a minimum of 5 times.

b) Spike each of the vessels with 10 µg mercury using the stock solution (4.2 j).

c) Follow the test procedure of interest in Clause 7 through the digestion and spectroscopic measurement.

d) Calculate the mercury concentration (µg/g) as indicated in Clause 9 and determine the per cent recovery of the spiked mercury for each of the samples.

$$SR = \frac{C \times M}{SA} \times 100$$

where

SR is the rate of recovery in % of the spiked mercury;

C is the measured concentration in µg/g;

M is the sample mass in g;

SA is the spike amount (10 µg).

The per cent recovery of mercury shall be between 80 % and 120 % for each of the samples. If the recovery is outside the limits for any of the replicates, the entire extraction and analysis procedure shall be repeated.

e) The method detection limit is obtained by calculating the standard deviation, *s*, for the replicate (minimum of 6) analyses. The standard deviation is then multiplied by Student's *t* value for the total number of replicates (*n*) for n-1 degrees of freedom. A list of Student's *t* values for 6 to 10 replicates is shown in Table 3.

EXAMPLE For 6 replicates and 6 – 1 = 5 degrees of freedom, the *t* value would be 3,36.

NOTE All analyses used to calculate an MDL should be consecutive.

Table 3 – Method detection limit = $t \times s_{n-1}$

Number of samples	Student's t-statistic 99 % confidence
6	3,36
7	3,14
8	3,00
9	2,90
10	2,82

f) The limit of quantification is determined by multiplying the method detection limit by a factor of 5.

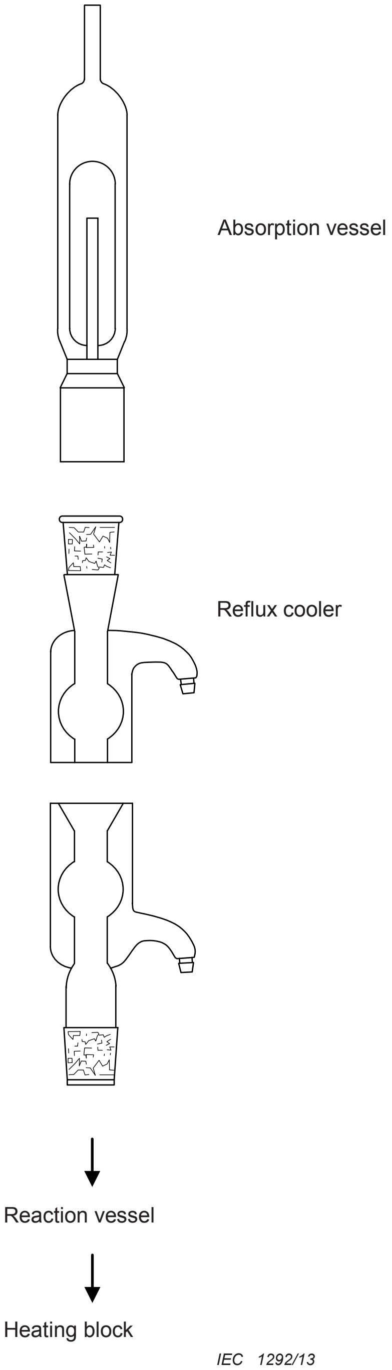
Method detection limits and limits of quantification will vary from laboratory to laboratory. Generally, a method detection limit of 2 µg/g (limit of quantification of 10 µg/g) has been found achievable using this method, however, this value depends on each instrument.

Annex A
(informative)

Practical application of determination of mercury in polymers, metals and electronics by CV-AAS, AFS, ICP-OES and ICP-MS

A.1 Equipment

Below is an example of the equipment used.



IEC 1292/13

Figure A.1 – Heating digester equipped with reaction vessel, reflux cooler and absorption vessel

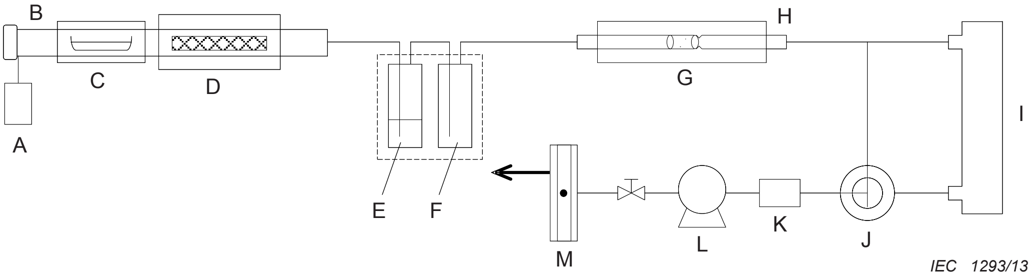
Table A.1 – Program for microwave digestion (example) of samples (power output for five vessels)

Step	**Time** min	**Power output** W	**Pressure limited to** MPa
1	5	400	3,5
2	5	600	3,5
3	12	800	3,5
4	20	800	4,0
5	3	500	4,0
Ventilation step	20	0	–

A.2 Thermal decomposition-gold amalgamation system

A.2.1 General

The system is made up of a mercury removing filter, a sample heating furnace, a decomposition furnace, a cooling dehumidifier, a mercury collector furnace, an absorption cell for cold-vapor atomic absorption spectrometry or fluorescence cell for cold-vapor fluorescence spectrometry, a switching valve, a suction pump, a flow meter, etc. Some of these factors, however, such as the matrix effect, dilution factor and interferences reposed method should be confirmed.



Key

A	mercury removal filter
B	combustion tube
C	sample heating furnace
D	decomposition furnace
E	gas-washing bottle
F	dehumidifier bottle
G	mercury collector furnace
H	mercury collecting tube
I	cold-vapor atomic absorption cell
J	switching valve
K	mercury remover
L	suction pump
M	flow meter

Figure A.2 – Configuration of equipment with AAS (example)

A.2.2 Carrier gas

Air is typically used as the carrier gas. Oxygen-rich gas can also be used as a carrier gas. Oxygen can produce explosion-like rapid combustion in the sample heating furnace, resulting in a large amount of combustion products instantly appearing, which can exceed the catalyzing capability of the downstream decomposition furnace, or part of the mercury generated in the decomposition may change into mercury oxide. The heating temperature and flow rate therefore need to be carefully selected. Ar gas is necessary specifically for the fluorescence spectrometry.

Any mercury in the carrier gas should be removed with a filter such as activated charcoal or gold mesh.

A.2.3 Heating unit

The combustion tube is made of ceramic or quartz. Sample vessels are made of heatproof materials. When liquid samples are analyzed, the vessel should be made of quartz or ceramic (mercury-free).

A.2.4 Decomposition furnace

A catalyst such as manganese oxide, copper oxide, cobalt oxide, or platinum is placed in the decomposition furnace within the combustion tube so as to decompose any organic substances and mercury compounds, thus requiring that the temperature and the carrier gas rate be carefully set in order to prevent mercury oxide being generated.

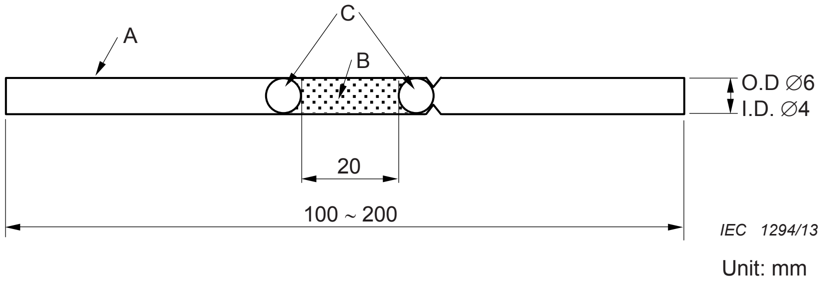
A.2.5 Washing and humidifying equipment

If the system has a cold spot in its plumbing and needs to dehumidify the moisture, cooled humidification involves easier handling and maintenance than humidifiers that use desiccant such as magnesium perchlorate. More effective results are also obtained when a gas-washing bottle is used before humidification takes place. Standard buffer solution should be used for gas washing.

A.2.6 Mercury collecting tube

An example of the mercury collecting tube is given in Figure A.3.

A quartz glass tube that is dimpled in the middle is filled with quartz glass wool, mercury collector, and quartz glass wool, in that order.



Unit: mm

Key

A quartz glass tube

B mecury collector

C quartz glass wool

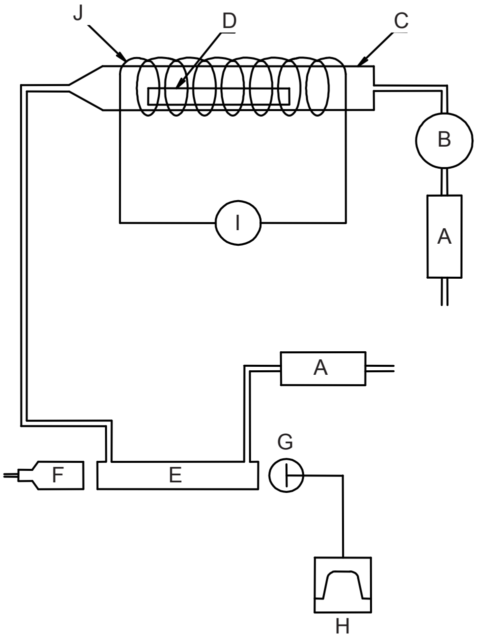
Figure A.3 – Mercury collecting tube (example)

A thermally stable, porous carrier (diatomaceous earth) coated with gold is used as the mercury collector. The mercury collecting furnace should be preheated to 150 °C to prevent the collection of organic gases and water by the mercury collector. Organic gases and water, if introduced into the absorption cell or the fluorescence cell, will interfere with the mercury measurement.

A.2.7 Mercury analysis for CCFL

In the case of a CCFL sample, the mercury vapour generating part generates mercury vapour by heating broken pieces of a CCFL lamp. The generated mercury vapour is then introduced into the atomic absorption spectrometer to measure the amount of mercury without amalgamation. The integrator begins to integrate the ultraviolet absorbance as soon as heating starts.

The configuration (example) of the thermal decomposition atomic absorption spectrometer for CCFL is shown in Figure A.4.



IEC 1295/13

Key

- A mercury removing device
- B air pump
- C quartz heating tube
- D quartz boat
- E quartz absorption cell
- F mercury lamp
- G atomic absorption detector
- H integrator
- I power unit with controller
- J heater

Figure A.4 – Configuration (example) of the thermal decomposition/atomic absorption spectrometer for CCFL

A.3 Instrument parameters

The listed instrument parameters are examples of workable instrument parameters and may differ, since individual instruments may require alternate parameters. The use of listed wavelengths and mass-charge ratios is highly recommended as the selection of other parameters in this context can cause false results.

a) CV-AAS

Light source: Electrodeless discharge lamp or hollow cathode lamp

Wavelength: 253,7 nm

Spectral bandwidth: 0,7 nm

Purge gas: N_2 or Ar

b) CV-AFS

Source: Hg hollow cathode lamp, current: 30 mA, wavelength: 253,7 nm

Minus high-voltage: 360 V

Oven temperature: 800 °C

Argon flow carrier gas: 0,6 l/min, shield gas: 1,0 l/min

Wash water: a mass fraction of 6 % HNO_3

c) ICP-OES

Hg wavelength: 194,227 × 2 (2nd order) nm

RF generator power: 1 150 W

Frequency of RF generator: 27,12 MHz

Argon pressure: 0,16 MPa

Argon flow carrier gas: Cool gas: 14 l/min, auxiliary gas: 0,5 l/min

Sample uptake rate: 1,6 ml/min

d) ICP-MS

Mass-charge ratios for Hg: $m/z = 199, 200, 201, 202$

RF generator power: 1 200 W

Frequency of RF generator: 27,12 MHz

Argon pressure: 0,28 MPa

Argon flow carrier gas: Cooling gas: 16 l/min, auxiliary gas: 1,0 l/min

All conditions such as torch position: sampling depth, horizontal and vertical alignment, as well as lenses should be optimized before measurement.

e) TD(G)-AAS

Light source: Low pressure mercury (vapour) lamp,

Wavelength: 253,7 nm

Carrier gas: air, or oxygen

Annex B
(informative)

Results of international interlaboratory study Nos. 2 (IIS2) and 4A (IIS 4A)

Table B.1 – Statistical data for TD(G)-AAS

Polymer Sample	**Parameter**	***m*** mg/kg	***v*** mg/kg	***n***	***s(r)*** mg/kg	***r*** mg/kg	***s(R)*** mg/kg	***R*** mg/kg
IIS4A-04	Hg	31,28	33	18	0,246	0,69	1,50	4,19
IIS4A-05	Hg	56,83	63	9	0,76	2,1	3,10	8,70
IIS4A-08	Hg	22,67	24	15	0,856	2,40	1,96	5,47

Table B.2 – Statistical data for CV-AAS

Polymer Sample	**Parameter**	***m*** mg/kg	***v*** mg/kg	***n***	***s*(*r*)** mg/kg	***r*** mg/kg	***s*(*R*)** mg/kg	***R*** mg/kg
IIS2-C10	Hg	21,4	25,3	3	2,5	7,0	Insufficient data	
IIS2-C11	Hg	4,2	4,5	2	0,1	0,4		
IIS2-C12	Hg	88,5	100,0	8	1,2	3,3	4,9	13,7
IIS2-C13	Hg	883,5	1 000	8	16,1	45,1	59,0	165,1
IIS4A-04	Hg	30,03	33	15	0,55	1,55	1,32	3,70
IIS4A-05	Hg	52,58	63	13	1,45	4,07	3,78	10,59

Table B.3 – Statistical data for CV-AFS

Polymer Sample	**Parameter**	***m*** mg/kg	***v*** mg/kg	***n***	***s*(*r*)** mg/kg	***r*** mg/kg	***s*(*R*)** mg/kg	***R*** mg/kg
IIS2-C10	Hg	24,8	25,3	3	0,2	0,6	Insufficient data	
IIS2-C11	Hg	4,8	4,5	3	0,2	0,4		
IIS4A-04	Hg	30,2	33	18	1,90	5,32	5,35	14,99
IIS4A-05	Hg	54,6	63	18	2,55	7,14	9,70	27,16

Table B.4 – Statistical data for ICP-OES

Polymer Sample	**Parameter**	***m*** mg/kg	***v*** mg/kg	***n***	***s*(*r*)** mg/kg	***r*** mg/kg	***s*(*R*)** mg/kg	***R*** mg/kg
IIS2-C10	Hg	25,4	25,3	6	2,6	7,2	6,8	19,2
IIS2-C11	Hg	3,8	4,5	3	0,5	1,4	Insufficient data	
IIS2-C12	Hg	90,7	100,0	9	3,1	8,8	8,2	23,1
IIS2-C13	Hg	901,7	1 000	9	41,5	116,2	68,8	192,7

where

m is the arithmetic mean of test results;
v is the expected value;
n is the number of accepted results;
$s(r)$ is the repeatability standard deviation;
r is the repeatability limit;
$s(R)$ is the reproducibility standard deviation;
R is the reproducibility limit.

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[3] To be published.

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