IEC 62321-5

Edition 1.0 2013-06

INTERNATIONAL STANDARD

NORME INTERNATIONALE

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HORIZONTAL STANDARD

NORME HORIZONTALE

Determination of certain substances in electrotechnical products – Part 5: Cadmium, lead and chromium in polymers and electronics and cadmium and lead in metals by AAS, AFS, ICP-OES and ICP-MS

Détermination de certaines substances dans les produits électrotechniques – Partie 5: Du cadmium, du plomb et du chrome dans les polymères et les produits électroniques, du cadmium et du plomb dans les métaux par AAS, AFS, ICP-OES et ICP-MS

IEC 62321-5:2013

IEC Central Office
3, rue de Varembé
CH-1211 Geneva 20
Switzerland

Tel.: +41 22 919 02 11
Fax: +41 22 919 03 00
info@iec.ch
www.iec.ch

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DETERMINATION OF CERTAIN SUBSTANCES IN ELECTROTECHNICAL PRODUCTS –

Part 5: Cadmium, lead and chromium in polymers and electronics and cadmium and lead in metals by AAS, AFS, ICP-OES and ICP-MS

FOREWORD

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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 62321-5 is a partial replacement of IEC 62321:2008, forming a structural revision and generally replacing Clauses 8 to 10, as well as Annexes F, G and H.

Future parts in the IEC 62321 series will gradually replace the corresponding clauses from 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
111/297/FDIS	111/307/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.

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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 (PBDE's)) 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 5: Cadmium, lead and chromium in polymers and electronics and cadmium and lead in metals by AAS, AFS, ICP-OES and ICP-MS

1 Scope

This Part of IEC 62321 describes the test methods for lead, cadmium and chromium in polymers, metals and electronics by AAS, AFS, ICP-OES and ICP-MS.

This standard specifies the determination of the levels of cadmium (Cd), lead (Pb) and chromium (Cr) in electrotechnical products. It covers three types of matrices: polymers/polymeric workpieces, metals and alloys and electronics.

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 AAS (atomic absorption spectrometry), AFS (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.

As the hexavalent-Cr analysis is sometimes difficult to determine in polymers and electronics, this standard introduces the screening methods for chrome in polymers and electronics except from AFS. Chromium analysis provides information about the existence of hexavalent-Cr in materials. However, elemental analyses cannot selectively detect hexavalent-Cr; it determines the amount of Cr in all oxidation states in the samples. If Cr amounts exceed the hexavalent-Cr limit, testing for hexavalent-Cr should be performed.

The test procedures described in this standard are intended to provide the highest level of accuracy and precision for concentrations of Pb, Cd and Cr that range, in the case of ICP-OES and AAS, from 10 mg/kg for Pb, Cd and Cr, in the case of ICP-MS, from 0,1 mg/kg for Pb and Cd in the case of AFS, the range is from 10 mg/kg for Pb and 1.5 mg/kg for Cd. The procedures are not limited for higher concentrations.

This standard does not apply to materials containing polyfluorinated polymers because of their stability. If sulfuric acid is used in the analytical procedure, there is a risk of losing Pb, thus resulting in erroneously low values for this analyte. In addition, sulfuric acid and hydrofluoric acid are not suitable for determining Cd by AFS, because it disturbs the reduction of Cd.

Limitations and risks occur due to the solution step of the sample, e.g. precipitation of the target or other elements may occur, in which case the residues have to be checked separately or dissolved by another method and then combined with the test sample solution.

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 certain substances in electrotechnical products – Part 1: Introduction and overview*[1]

IEC 62321-2, *Determination of certain substances in electrotechnical products – Part 2: Disassembly, disjointment and mechanical sample preparation*[1]

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

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

ISO 5961, *Water quality – Determination of cadmium by atomic absorption spectrometry*

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
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)

3.1.3
calibration solution
solution used to calibrate the instrument prepared either from (a) stock solution(s) or from a (certified) reference material

3.1.4
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 procedures

3.1.5
laboratory control sample
known matrix spiked with compound(s) representative of the target analytes, used to document laboratory performance

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1 To be published.

[Based on US EPA SW-846] [1] [2]

3.1.6
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)

3.1.7
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

CCV continuing calibration verification

LCS laboratory control sample

4 Reagents

4.1 General

For the determination of elements at trace level, the reagents shall be of adequate purity. The concentration of the analyte or interfering substances in the reagents and water shall be negligible compared to the lowest concentration to be determined.

All reagents for ICP-MS analysis, including acids or chemicals used shall be of high-purity: trace metals shall be less than 1×10^{-6} % in total.

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

4.2 Reagents

The following reagents are used:

a) Water: Grade 1 specified in ISO 3696 used for preparation and dilution of all sample solutions.

b) Sulfuric acid:
   1) Sulfuric acid: $\rho(H_2SO_4)$ = 1,84 g/ml, a mass fraction of 95 %, “trace metal” grade.
   2) Sulfuric acid: dilution (1:2): dilute 1 volume of concentrated sulfuric acid (4.2 b 1)) with 2 volumes of water (4.2 a))

c) Nitric acid:
   1) Nitric acid: $\rho(HNO_3)$ = 1,40 g/ml, a mass fraction of 65 %, “trace metal” grade.
   2) Nitric acid, a mass fraction of 10 %, “trace metal” grade.
   3) Nitric acid: 0,5 mol/l, “trace metal” grade.
   4) Nitric acid: dilution (1:2): dilute 1 volume of concentrated nitric acid (4.2.c 1)) with 2 volumes of water (4.2 a))

d) Hydrochloric acid:
   1) Hydrochloric acid, $\rho(HCl)$ = 1,19 g/ml, a mass fraction of 37 %, “trace metal” grade.

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2 Figures in square brackets refer to the Bibliography.

  2) Hydrochloric acid: dilution (1:2): dilute 1 volume of concentrated hydrochloric acid (4.2.d) 1)) with 2 volumes of water (4.2 a))
  3) Hydrochloric acid, a mass fraction of 5 %, “trace metal” grade.
  4) Hydrochloric acid, a mass fraction of 10 %, “trace metal” grade.

e) Hydrofluoric acid: ρ(HF) = 1,18 g/ml, a mass fraction of 40 %, “trace metal” grade.

f) Fluoroboric acid: HBF_4, a mass fraction of 50 %, “trace metal” grade.

g) Perchloric acid: ρ($HClO_4$) =1,67 g/ml, a mass fraction of 70 %, “trace metal” grade.

h) Phosphoric acid: ρ(H_3PO_4) =1,69 g/ml, more than a mass fraction of 85 %, “trace metal” grade.

i) Hydrobromic acid: ρ(HBr) = 1,48 g/ml, a mass fraction of 47 % to 49 %, “trace metal” grade.

j) Boric acid (H_3BO_3): 50 mg/ml, a mass fraction of 5 %, “trace metal” grade.

k) Hydrogen peroxide: ρ(H_2O_2) = 1,10 g/ml, a mass fraction of 30 %, “trace metal” grade.

l) Mixed acid:
  1) Mixed acid 1, two parts hydrochloric acid (4.2 d) 1)), one part nitric acid (4.2 c)1)) and two parts water (4.2 a)).
  2) Mixed acid 2, one part nitric acid (4.2 c) 1)) and three parts hydrofluoric acid (4.2 e)).
  3) Mixed acid 3, three parts hydrochloric acid (4.2 d) 1)) and one part nitric acid (4.2 c)1)).

m) Potassium hydroxide (KOH), “trace metal” grade.

n) Potassium borohydride (KBH_4), “trace metal” grade.

o) Potassium ferricyanide ($K_3(Fe(CN)_6)$), “trace metal” grade.

p) Oxido – reduction agent: a mass fraction of 1,5 % KBH_4 – a mass fraction of 1 % $K_3(Fe(CN_6)$ in a mass fraction of 0,2 % KOH.

  Add approximately 800 ml of water (4.2 a)) to a 1 000 ml volumetric flask (5.2 e)3)) followed by the addition of 2 g potassium hydroxide (4.2 m)). Add 15 g potassium borohydride (4.2 n)) and 10 g potassium ferricyanide (4.2 o)), stir to dissolve. Fill up to the mark with water (4.2 a)). Prepare daily.

q) Reducing agents:
  1) Reducing agent 1, a mass fraction of 3 % KBH_4 in a mass fraction of 0,2 % KOH:

     Add approximately 800 ml of water (4.2 a)) to a 1 000 ml volumetric flask (5.2 e) 3)) followed by the addition of 2 g potassium hydroxide (4.2 m)). Add 30 g of potassium borohydride (4.2 n)), stir to dissolve. Fill up to the mark with water (4.2 a)). Prepare daily.

  2) Reducing agent 2, a mass fraction of 4 % KBH_4 in a mass fraction of 0,8 % KOH.

     Add approximately 800 ml of water (4.2 a)) to a 1 000 ml volumetric flask (5.2 e) 3)), followed by the addition of 8 g potassium hydroxide (4.2 m)). Add 40 g of potassium borohydride (4.2 n)), stir to dissolve. Fill up to the mark with water (4.2 a)). Prepare daily.

r) Carrier flow:
  1) Carrier flow 1, a mass fraction of 1,5 % HCl.
  2) Carrier flow 2, a mass fraction of 1 % HCl.

s) Thiourea ($(NH_2)_2CS$) solution, a mass fraction of 10 % . Prepare daily.

t) Masking agent:
  1) Masking agent 1, a mass fraction of 5 % oxalic acid – a mass fraction of 5 % potassium sulfocyanate (KSCN) – a mass fraction of 0,5 % o-phenanthroline ($C_{12}H_8N_2$) solution:

     Add 10 g oxalic acid, 10 g potassium sulfocyanate and 1 g o-phenanthroline to 200 ml of water (4.2 a)). Heat at low temperature and stir to dissolve, taking care to avoid

boiling of the solution. Use the solution before the solid crystallizes out. Discard the solution when it becomes dark and prepare a fresh one.

2) Masking agent 2, a mass fraction of thiourea 10 % – ascorbic acid a mass fraction of 10 % solution.

Dissolve 10 g thiourea and 10 g ascorbic acid in 100 ml of water. Prepare daily.

u) Cobalt solution, 50 mg/l.

v) Stock solution:

1) Stock solution with 1 000 mg/l of Pb.

2) Stock solution with 1 000 mg/l of Cd.

3) Stock solution with 1 000 mg/l of Cr.

4) Stock solution with 10 000 mg/l of Fe.

5) Stock solution 10 000 mg/l of Cu.

w) Internal standard stock solution.

1) 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.

2) For use with ICP-OES, Sc or Y is recommended. The recommended concentration is 1 000 mg/l.

3) For use with ICP-MS, Rh is recommended. The recommended concentration is 1 000 μg/l.

The toxicity of each reagent in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals at the lowest possible level by whatever means available is recommended.

Preparation methods involve the use of strong acids, which are corrosive and cause burns. Laboratory coats, gloves and safety glasses should be worn when handling acids.

Nitric acid gives off toxic fumes. Always carry out digestion in a fume cupboard, and also when adding acid to samples because of the possibility of toxic gases being released.

The exhaust gases from the plasma should be ducted away by an efficient fume extraction system.

Special precautionary measures should be taken when hydrofluoric acid is used, i.e. HF antidote gel (2,5 % calcium gluconate in a water-soluble gel) for first aid treatment of HF burns on the skin.

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

5 Apparatus

5.1 General

In general, the collection and storage of glassware are critical parts of trace analysis, regardless of the type of sample to be analysed. Because of the sensitivity of the Pb, Cd and Cr analysis techniques described, each individual sampling step shall be carried out with great care. All sampling, storage and manipulation apparatus shall be metal-free. Soak all glassware in 10 % nitric acid (4.2 c) 2)) for 24 h at room temperature, and then rinse thoroughly with water (4.2 a)).

5.2 Apparatus

The following equipment shall be used:

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

b) HF-resistant sample introduction system: system in which the sample insertion section and torch have been treated for resistance to HF.

c) Argon gas: gas with purity of over 99,99 %.

d) Acetylene gas: gas with purity of over 99,99 %.

e) Glassware: all glassware shall be cleaned with 10 % nitric acid (4.2 c) 2)) before use:

   1) Kjeldahl flask: 100 ml;

   2) Beakers: such as 100 ml, 200 ml, 500 ml etc.;

   3) Volumetric flasks: such as 50 ml, 100 ml, 200 ml, 500 ml, 1 000 ml, etc. Where appropriate, other types of volumetric equipment with acceptable precision and accuracy can be used as an alternative to volumetric flasks.

   4) Pipettes: such as 1 ml, 5 ml, 10 ml, 20 ml, etc.;

   5) Watch glass.

f) Crucibles of platinum: such as 50 ml, 150 ml, etc.

g) Crucibles of porcelain: such as 50 ml, 150 ml, etc.

h) PTFE/PFA equipment (polytetrafluoroethylene (PTFE)/perfluoro alkoxy alkane resin (PFA): all equipment shall be cleaned with 10 % nitric acid (4.2 c) 2)) before use:

   1) Beakers: such as 100 ml, 200 ml, 500 ml etc.;

   2) Covers for breakers;

   3) Volumetric flasks: such as 100 ml, 200 ml, 500 ml, etc.

i) Micropipettes: such as 10 µl, 100 µl, 200 µl, 500 µl, 1 000 µl etc.

j) Containers: for storage of standard solution and calibrant.

   Containers to be made of high-density polyethylene (PE-HD) or PFA bottles.

k) For determination at the ultra-trace level, containers made of perfluoro alkoxy alkane resin (PFA) or perfluoro (ethylene-propylene) plastic (FEP) shall be used. In either case, the user shall confirm the suitability of the container selected.

l) Electric hot plate or heated sand bath.

m) Muffle furnace: capable of being maintained at 550 °C ± 25 °C.

n) Bunsen burner or similar type of gas burner.

o) Digestion with aqua regia: digestion apparatus equipped with a time and temperature microcontroller unit, a heating block thermostat, a set of vessels, each equipped with reflux coolers and absorption vessels.

p) Microwave digestion system equipped with a sample holder and high-pressure polytetrafluoroethylene/tetrafluoroethylene modified (PTFE/TFM) or perfluoro alkoxy alkane resin/tetrafluoroethylene modified (PFA/TFM) or other vessels based on fluorocarbon materials.

   There are many safety and operational recommendations specific to the model and manufacturer of the microwave equipment used in individual laboratories. The analyst is required to consult the specific equipment manual, manufacturer and literature for proper and safe operation of the microwave equipment and vessels.

q) Heat-resistant thermal insulation board.

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

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

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

u) Atomic absorption spectrometer (AAS).

v) Atomic fluorescence spectrometer (AFS).

6 Sampling

6.1 General

The different test methods, which can be used as alternatives according to this International Standard, need different amounts of sample to obtain the required quality of results. Generally it is advisable to start with the highest amount of sample suitable for the chosen procedure.

In the case of electronics, the sample shall first be destroyed mechanically by appropriate means (e.g. grinding, milling, mill cutting) 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).

It is recommended to analyse aqueous sample solutions directly after sample preparation. If this is not possible, it is highly recommended to stabilize the solutions in an adequate way, and to store the solutions no longer than 180 days at ambient temperature.

6.2 Test portion

6.2.1 Polymers

For acid digestion, weigh 400 mg of sample that has been ground, milled or cut to the nearest 0,1 mg. For the dry ashing method, or for microwave digestion method, weigh 200 mg of sample that has been ground, milled or cut is measured to the nearest 0,1 mg.

6.2.2 Metals

Weigh 1 g of sample to the nearest 0,1 mg and is placed in a glass beaker or a PTFE/PFA beaker (5.2 h) 1)) when using HF (4.2 e)). For AFS, the quantity of the sample measured is 0,2 g.

6.2.3 Electronics

For digestion with aqua regia, weigh 2 g of the ground sample (maximum particle size: 250 µm) to the nearest 0,1 mg level. For microwave digestion method, weigh 200 mg of ground sample (maximum particle size: 250 µm) to the nearest 0,1 mg.

7 Procedure

7.1 Polymers

7.1.1 General

The samples are pre-cut and/or milled to an appropriate size for the method selected according to the procedure described in Clause 6. Depending on the particular method of preparing the test solution, sample amounts may vary, as described in detail in this clause. The test solution may be prepared by dry ashing or by sample digestion with acids such as nitric acid or sulfuric acid. Acid digestion can be carried out in a closed system using a microwave digestion vessel. Depending on the presence of particular elements, the details of the approach to digestion varies – procedures are given in this clause. Information on the presence of these elements may have been gained from previous screening experiments (IEC 62321-3-1). Finally, in the digestion solution obtained, Pb, Cd and Cr are determined by ICP-OES, ICP-MS or by AAS. In the case of AFS, before determination the digestion solution should be treated additionally for Pb and Cd.