IEC 62321-7-2

Edition 1.0 2017-03

INTERNATIONAL STANDARD

NORME INTERNATIONALE

HORIZONTAL STANDARD

NORME HORIZONTALE

Determination of certain substances in electrotechnical products – Part 7-2: Hexavalent chromium – Determination of hexavalent chromium (Cr(VI)) in polymers and electronics by the colorimetric method

Détermination de certaines substances dans les produits électrotechniques – Partie 7-2: Chrome hexavalent – Détermination du chrome hexavalent (Cr(VI)) dans les polymères et les produits électroniques par méthode colorimétrique

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DETERMINATION OF CERTAIN SUBSTANCES IN ELECTROTECHNICAL PRODUCTS –

Part 7-2: Hexavalent chromium – Determination of hexavalent chromium (Cr(VI)) in polymers and electronics by the colorimetric method

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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 62321-7-2 is a partial replacement of IEC 62321:2008, forming a structural revision and generally replacing Annex C. IEC 62321-7-2 is the final replacement part of the corresponding clauses in IEC 62321:2008.

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

CDV	Report on voting
111/408/CDV	111/432/RVC

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

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

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

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

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INTRODUCTION

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

The use of hexavalent chromium in electrotechnical products is of concern in many regions of the world.

The purpose of this document is therefore to provide test methods that will allow the electrotechnical industry to determine the levels of hexavalent chromium in electrotechnical products on a consistent global basis.

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

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DETERMINATION OF CERTAIN SUBSTANCES IN ELECTROTECHNICAL PRODUCTS –

Part 7-2: Hexavalent chromium – Determination of hexavalent chromium (Cr(VI)) in polymers and electronics by the colorimetric method

1 Scope

This part of IEC 62321 describes procedures to measure hexavalent chromium, Cr(VI), quantitatively in samples of polymers and electronics. This method employs organic solvent to dissolve or swell the sample matrix, followed by an alkaline digestion procedure to extract Cr(VI) from samples. Studies have shown that organic/alkaline solution is more effective than acidic solution in extracting Cr(VI) from soluble and insoluble samples. Minimal reduction of Cr(VI) to Cr(III) or oxidation of Cr(III) to Cr(VI) occurs under alkaline conditions.

For soluble polymers consisting of ABS (Acrylonitrile- butadiene-styrene), PC (Polycarbonate) and PVC (poly(vinyl chloride)), the samples are first dissolved in an appropriate organic solvent and Cr(VI) is then extracted by an alkaline extraction solution.

For insoluble/unknown polymers, or electronic materials that do not contain antimony (Sb), the samples are digested in a toluene/alkaline solution at 150 °C to 160 °C. Then the organic phase in the extracts are separated and discarded; the inorganic phase is retained for Cr(VI) analysis.

The Cr(VI) concentration in the extract is determined by its reaction under acidic conditions with 1,5-diphenylcarbazide. Cr(VI) is reduced to Cr(III) in the reaction with diphenylcarbazide which is oxidized to diphenylcarbazone. The Cr(III) and diphenylcarbazone form a red-violet-coloured complex in the reaction. The complex solution is measured quantitatively by a colorimeter or a spectrophotometer at 540 nm.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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*

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

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

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

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- ISO Online browsing platform: available at http://www.iso.org/obp

3.2 Abbreviated terms

For the purposes of this document, the abbreviated terms given in IEC 62321-1 apply.

4 Reagents

4.1 General

Use only reagents of recognized analytical grade, unless otherwise specified.

4.2 Reagents

The following reagents shall be used:

a) N-Methyl-pyrrolidone (NMP), analytical reagent grade. Add 10 g activated molecular sieves (4.2 r)) per 100 ml of newly opened NMP, seal the cap tightly, keep in the dark, shake occasionally and maintain over 12 h before first use. Store at 20 °C to 25 °C with molecular sieves in a tightly sealed brown glass container and avoid direct light exposure. The suggested maximum storage period is four weeks after the opening time of the container.

b) Nitric acid, volume fraction of 35 %. Dilute 50 ml of reagent grade HNO_3 to 100 ml with water (see 4.2 p)) in a volumetric flask (5.2 j)). Store at 20 °C to 25 °C in the dark. Do not use concentrated HNO_3 if it has a yellow colour, which is an indication of photoreduction of NO_3^- to NO_2^-, a reducing agent for Cr(VI).

c) Sodium carbonate: Na_2CO_3, anhydrous, analytical reagent grade.

d) Sodium hydroxide: NaOH, analytical reagent grade.

e) Magnesium chloride: $MgCl_2$ (anhydrous), analytical reagent grade. A mass of 200 mg $MgCl_2$ is approximately equivalent to 50 mg Mg^{2+}.

f) Phosphate buffer. To prepare a buffer solution at pH 7, dissolve 87,09 g K_2HPO_4 (analytical reagent grade) and 68,04 g KH_2PO_4 (analytical reagent grade) into 700 ml of water (4.2 p)). Transfer to a 1 l volumetric flask (5.2 j)) and dilute to volume. As prepared, the solution will contain 0,5 mol/l K_2HPO_4 and 0,5 mol/l KH_2PO_4.

g) Lead chromate: $PbCrO_4$, analytical reagent grade. Store at 20 °C to 25 °C in a tightly sealed container. This is the agent used for the matrix spike recovery correction method.

h) Digestion solution. Dissolve 20,0 g ± 0,05 g NaOH and 30,0 g ± 0,05 g Na_2CO_3 in water (4.2 p)) in a 1 l volumetric flask (5.2 j)) and dilute to the mark. Store the solution in a tightly capped polyethylene bottle at 20 °C to 25 °C, and prepare fresh monthly. The pH of the digestion solution shall be checked before using. If the pH is $< 11,5$ discard the solution and prepare a fresh batch.

i) Toluene, analytical reagent grade.

j) Potassium dichromate stock solution. Dissolve 141,4 mg of dried (105 °C) $K_2Cr_2O_7$ (analytical reagent grade) in water (4.2 p)) and dilute to 1 l in a volumetric flask (5.2 j)) (1 ml contains 50 μg Cr).

k) Potassium dichromate standard solution. Dilute 10 ml potassium dichromate stock solution (4.2 j)) with water (4.2 p)) to 100 ml in a volumetric flask (5.2 j)) (1 ml contains 5 μg Cr).

l) Sulfuric acid, volume fraction of 10 %. Dilute 10 ml of distilled reagent grade or spectroscopic grade H_2SO_4 to 100 ml with water (4.2 p)) in a volumetric flask (5.2 j)).

m) Diphenylcarbazide solution. Dissolve 250 mg 1,5-diphenylcarbazide in 50 ml acetone (4.2 q)). Store in a brown bottle. Prior to use, check the solution for discoloration. Store for

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use up to two weeks and if solution becomes discoloured, discard it and prepare a fresh batch.

n) Potassium dichromate, $K_2Cr_2O_7$, spike solution 1 000 mg/l Cr(VI)). Dissolve 2,829 g of dried (105 °C) $K_2Cr_2O_7$ in water (4.2 p)) in a 1 l volumetric flask (5.2 j)), and dilute to the mark. Alternatively, a 1 000 mg/l Cr(VI)-certified standard solution can be used. Store for use up to six months at 20 °C to 25 °C in a tightly sealed container.

o) Potassium dichromate, $K_2Cr_2O_7$, matrix spike solution (100 mg/l Cr(VI)): Add 10,0 ml of the 1 000 mg/l Cr(VI) solution made from $K_2Cr_2O_7$ spike solution (4.2 n)) to a 100 ml volumetric flask (5.2 j)) and dilute to volume with water (4.2. p)). Mix well.

p) Water. Grade 3 specified in ISO 3696, which shall be free of interferences.

q) Acetone, analytical reagent grade.

r) Molecular sieves (4A), CAS: 70955-01-0, desiccant.

WARNING – All potential Cr(VI)-containing samples and reagents used in the method have to be handled with appropriate precautions. Solutions or waste material containing Cr(VI) have to be disposed of properly. For example, ascorbic acid or some other reducing agents can be used to reduce Cr(VI) to Cr(III).

5 Apparatus

5.1 General

All re-usable laboratory ware (glass, quartz, polyethylene, polytetrafluoroethylene (PTFE), etc.) including the sample containers shall be soaked overnight in laboratory-grade detergent and water, rinsed with water, and soaked for 4 h or more in HNO_3 (volume fraction of 20 %) or in a mixture of dilute acids (HNO_3:HCl:H_2O = 1:2:9 by volume) followed by rinsing with water (4.2 p)). Alternative cleaning procedures are permitted, provided adequate cleanliness can be demonstrated through the analysis of method blanks.

5.2 Apparatus

The following items shall be used for the analysis:

a) Vacuum filtration apparatus.

b) Heating or microwave device capable of maintaining the digestion solution at temperatures between 150 °C and 160 °C.

c) Ultrasonic water bath, capable of maintaining the temperature between 60 °C and 65 °C.

d) Calibrated pH meter to read pH in a range of 0 to 14 with an accuracy of ±0,03 pH units.

e) Analytical balance capable of measurement to 0,1 mg.

f) Thermometer, thermistor or other temperature measurement device capable of measuring up to 160 °C.

g) Colorimetric instrument: either a spectrophotometer for use at 540 nm, providing a light path of 1 cm or longer or a filter photometer, providing a light path of 1 cm or longer and equipped with a filter having maximum transmittance near 540 nm.

h) Grinding mill, with or without liquid nitrogen cooling, capable of grinding polymer samples and electronic components.

i) Borosilicate glass or quartz beaker with volume graduation of 150 ml, or equivalent.

j) Volumetric glassware: Class A or equivalent of acceptable precision and accuracy. Alternative volumetric equipment (e.g. automatic dilutors) with equivalent precision and accuracy can be used.

k) Assorted calibrated pipettes: Class A glassware or other with equivalent precision and accuracy.

l) Digestion vessel: Glass screw thread bottle (wide neck), volume of 50 ml and minimum inner diameter of 3 cm.

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m) Glass separatory funnel, 100 ml.

n) Filter membranes (0,45 μm): preferably cellulose-based or PC membranes; filter syringe (0,45 μm): nylon or PVDF.

o) C18 syringe filter cartridge.

p) Microwave digestion vessel or a suitable borosilicate glass or quartz vessel equipped with a membrane for pressure relief over 1,0 MPa and volume graduation of 50 ml or equivalent.

6 Sampling

Samples shall be collected and stored using devices and containers that do not contain stainless steel.

For soluble polymers (ABS, PC and PVC), a particle size larger than 250 µm is acceptable, however, a longer dissolution time may be required to completely dissolve the polymer matrix.

Insoluble or unknown polymers and electronic components that do not contain Sb shall be ground into a fine powder (5.2. h)) prior to digestion to promote extraction, with 100 % of the material passing through a 250 μm sieve, for example a No. 60 ASTM standard sieve.

If the identity of the polymer matrix is unknown, a solubility test can be performed by testing a small amount of the sample using an organic solvent. Alternatively, infrared spectroscopy (IR) can be performed to identify the bulk polymer. The presence of Sb can be detected by X-ray fluorescence spectroscopy (XRF).

7 Test procedure

7.1 Extraction of Cr(VI) in soluble polymers – ABS, PC and PVC matrixes

a) Accurately weigh a sample of 0,1 g. Place the sample into a digestion vessel (5.2. l)).

NOTE Alternative sample amounts can be used for samples with potentially very low or very high Cr(VI) concentrations.

b) Place 10 ml of NMP (4.2. a)) into the digestion vessel (5.2. l)) and seal the cap tightly.

c) Dissolve each polymer sample by ultrasonication (5.2 c)) at 60 °C for 1 h. Shake the sample vessel by hand for about 10 s to suspend insoluble particles, then ultrasonicate at 60 °C for 1 h again. The sample matrix has to be completely dissolved before proceeding to the next step.

d) To test for recovery in every matrix, accurately weigh a second sample of 0,1 g (or another chosen amount of sample). Place it into the digestion vessel (5.2. l)) and add 10 ml of NMP (4.2 a)) into the vessel and seal the cap tightly. Then proceed with step 7.1 c), choose a matrix spike solution (4.2 o)) and add it directly to the sample. Follow steps from 7.1 e) to 7.1 o).

e) Shake the digestion vessel by hand and mix well, then add 200 mg $MgCl_2$ (4.2 e)) and 0,5 ml of 0,5 mol/L phosphate buffer (4.2 f)) to each digestion vessel. Shake the digestion vessel again and mix well.

f) Measure 20 ml of the digestion solution (4.2 h)) using a graduated cylinder (5.2 j)) and slowly pour into each digestion vessel (5.1 l)). Mix well.

g) Ultrasonicate above solution at 60 °C for 1 h (shake the digestion vessel by hand and mix well after 0,5 h).

h) Transfer above content to a 150 ml beaker (5.2 i)). With constant stirring while monitoring the pH, add HNO_3 (4.2 b)) dropwise to the beaker. Adjust the pH of the solution to 7,5 ± 0,5.

i) Even if the sample solution is turbid or flocculent precipitates are present, do not filter the sample solution.

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j) Add 2,5 ml diphenylcarbazide solution (4.2 m)) to each vessel. Slowly add H_2SO_4 solution (4.2 l)) to the vessel and adjust the pH of the solution to 2,0 ± 0,5.

k) Transfer the contents of the vessel quantitatively to a 100 ml volumetric flask (5.2 j)) and make up to the mark by water (4.2 p)). Mix well.

l) Filter the coloured sample solution using the 0,45 µm syringe filter (5.2 n)).

m) Transfer an appropriate portion of the solution to a 1 cm absorption cell and measure its absorbance at 540 nm with a colorimetric instrument (5.2 g)). Measurement shall be taken within 30 min of colour development.

n) Correct the absorbance reading of the sample by subtracting the absorbance of a blank carried through the colour development procedures.

o) From the corrected absorbance, determine the concentration of Cr(VI) present by referring to the calibration curve.

7.2 Extraction of Cr(VI) in insoluble/unknown polymers and electronics – without Sb

a) Accurately weigh a sample of 0,15 g. Place the sample into a digestion vessel (5.2 p)).

NOTE Alternative sample amounts can be used for samples with potentially very low or very high Cr(VI) concentrations.

b) To each sample add 10 ml of digestion solution (4.2 h)) and 5 ml of toluene (4.2 i)) measured with a graduated cylinder (5.2 j)).

c) To test for recovery in every matrix, accurately weigh a second sample of 0,15 g and place it into a second digestion vessel (5.2 p)). Choose a spike solution (4.2 n) or 4.2 o)) and add it directly to the sample. Add 10 ml of digestion solution (4.2 h)) and 5 ml of toluene (4.2 i)) measured with a graduated cylinder (5.2 j)).

d) Next, add 400 mg $MgCl_2$ (4.2 e)) and 0,5 ml of 1,0 mol/L phosphate buffer (4.2 f)) to each sample and mix well.

$MgCl_2$ is added to the solution to correct possible oxidation/reduction of chromium that may be caused by the analytical method.

e) Heat each sample to a temperature of 150 °C to 160 °C in a closed digestion vessel (5.2 p)) using a microwave oven or suitable heating device (5.2 b)). Then maintain temperature at 150 °C to 160 °C for 1,5 h and allow sample to cool to room temperature.

f) Separate the organic phase from the vessel using a separatory funnel (5.2 m)) and discard it. The aqueous phase is filtered through a 0,45 µm membrane filter (5.2 n)). Rinse the digestion vessel (5.2 p)) three times with water (4.2 p)) and filter. If the filter becomes clogged using the 0,45 µm membrane filter; a large pore size filter paper may be used to filter the samples.

g) Rinse the inside of the filter flask and the filter pad with water (4.2 p)) and transfer the filtrate and the rinse solutions to a clean 150 ml beaker (5.2. i)).

h) With constant stirring while monitoring the pH, add HNO_3 (4.2 b)) dropwise to the vessel obtained in 7.2 g). Adjust the pH of the solution to 7,5 ± 0,5.

i) If the sample solution is clear after pH adjustment, add 2,5 ml diphenylcarbazide solution (4.2 m)) to each vessel. Slowly add H_2SO_4 solution (4.2 l)) to the vessel and adjust the pH of the solution to 2,0 ± 0,5. Proceed with step 7.2 l). If the solution is turbid or contains a flocculent precipitate (cloudy, flake-like and non-crystalline), or colour is present, proceed to 7.2 j).

j) If the solution is turbid or flocculent precipitates are present, filter the sample through a 0,45 µm membrane filter (5.2 n)). If colour is present in the sample solution, filter the solution with a C18 syringe cartridge (5.2 o)) before adding diphenylcarbazide solution (4.2 m)). If the digestate is clear after either filtration step, add 2,5 ml diphenylcarbazide solution (4.2 m)) to the vessel. Slowly add H_2SO_4 solution (4.2 l)) to the vessel and adjust the pH of the solution to 2,0 ± 0,5. Proceed to 7.2 l). If the digestate is coloured or turbid after either filtration step, slowly add H_2SO_4 solution (4.2 l)) to the vessel and adjust the pH of the solution to 2,0 ± 0,5. Proceed to 7.2.k).

k) Transfer each of the coloured or turbid digestates quantitatively to a 50 ml volumetric flask (5.2 j)) and adjust to volume with water (4.2 p)). Invert several times to mix. Remove

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