INTERNATIONAL STANDARD

ISO 8124-5

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Safety of toys —

Part 5: Determination of total concentration of certain elements in toys

Sécurité des jouets —

Partie 5: Détermination de la concentration totale de certains éléments dans les jouets

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

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The committee responsible for this document is ISO/TC 181, *Safety of toys*.

ISO 8124 consists of the following parts, under the general title *Safety of toys*:

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— *Part 1: Safety aspects related to mechanical and physical properties*

— *Part 2: Flammability*

— *Part 3: Migration of certain elements*

— *Part 4: Swings, slides and similar activity toys for indoor and outdoor family domestic use*

— *Part 5: Determination of total concentration of certain elements in toys*

— *Part 6: Certain phthalate esters in toys and children's products*

— *Part 7: Requirements and test methods for finger paints*

— *Part 8: Age determination guidelines*

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Introduction

See A.1 (use and applicability).

This part of ISO 8124 defines a method for the determination of the total concentration of certain elements in toy materials and can be used to decide whether there is a need to undertake migration testing in accordance with the method specified in ISO 8124-3, *Migration of certain elements* or other equivalent standards, e.g. EN 71-3:1994/AC:2002 or ASTM F963-11. A material can be considered to conform to the requirements of ISO 8124-3:2010 if the total concentration results are below the soluble limits as prescribed in ISO 8124-3:2010, Table 1. If the soluble limits in ISO 8124-3:2010, Table 1 are exceeded, migration testing in accordance with ISO 8124-3:2010 will be required to determine compliance with ISO 8124-3:2010.

In addition, decisions can be also taken, within the scope of this part of ISO 8124, on the compliance of the material with any regulatory requirements that impose restrictions on the total concentration of certain elements.

Where legal conformity requires migration testing, this part of ISO 8124 can only be used to non-quantitatively confirm compliance with regulatory limits.

Users of this part of ISO 8124 are reminded that it has been developed only for the eight elements listed in Table 1. The use of this method for other elements must be validated by the user.

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Safety of toys —

Part 5: Determination of total concentration of certain elements in toys

1 Scope

1.1 This part of ISO 8124 specifies methods of sampling and digestion prior to analysis of the total concentration of the elements antimony, arsenic, barium, cadmium, chromium, lead, mercury, and selenium from toy materials and from parts of toys.

NOTE Other elements can be determined by this method provided adequate analytical performance is demonstrated. Manufacturers are encouraged to apply the test methods of this part of ISO 8124 and the limits from ISO 8124-3 to raw materials used in the manufacture of toys to give increased certainty of conformity to the requirements of ISO 8124-3.

1.2 Digestion methods for the elements mentioned in 1.1 are specified for the following types of toy materials:

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— coatings of paints, varnishes, lacquers, printing inks, polymers, and similar coatings;

— polymeric and similar materials, including laminates, whether textile-reinforced or not, but excluding other textiles;

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— paper, paperboard, and cardboard;

— natural or synthetic textiles;

— metallic materials whether coated or not;

— other materials, whether mass-coloured or not (e.g. wood, fibreboard, hardboard, bone, and leather);

— materials intended to leave a trace (e.g. the graphite materials in pencils and liquid ink in pens);

— pliable modelling materials, including modelling clays and gels;

— paints to be used as such in the toy, including finger paints, varnishes, lacquers, and similar materials in solid or liquid form;

— packaging materials that form part of the toy or have intended play value (see A.2.1, packaging).

NOTE Digestion methods for glass, ceramic, and other siliceous materials or fluorinated polymers or fluorinated polymer coatings are not described, and these types of materials are outside the scope of this part of ISO 8124 (see A.1, use and applicability).

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.

ISO 8124-1, *Safety of toys — Part 1: Safety aspects related to mechanical and physical properties*

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ISO 8124-3, *Safety of toys — Part 3: Migration of certain elements*

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
base material
material upon which *coatings* (3.2) can be formed or deposited

3.2
coating
all layers of material formed or deposited on the *base material* (3.1)of a toy, including paints, varnishes, lacquers, inks, polymers, or other substances of a similar nature, whether they contain metallic particles or not, no matter how they have been applied to the toy

Note 1 to entry: This definition includes metallic coatings deposited on a metal surface such as an electroplated coating. However, electroplating will only require testing if it can be removed by *scraping* (3.8); otherwise, it may be tested with the base material.

3.3
complete digestion
complete breakdown of the original material leaving only insoluble residues

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3.4
composite test portion
test portion (3.9) that is composed of more than one similar material type or colour of material

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3.5
detection limit of instrument
three times the standard deviation of the result obtained in the blank test using a specific instrument

3.6
laboratory sample
toy either in the form in which it is marketed, or in the form in which it is intended to be marketed

3.7
sample blank
solution that has undergone the same digestion processes used for the digestion of *test portions* (3.9) and consists of all reagents excluding the test portion

3.8
scraping
mechanical process for removal of *coatings* (3.2) down to the *base material* (3.1) using a sharp blade such as a scalpel

3.9
test portion
single material taken from an accessible part of a *laboratory sample* (3.6)

Note 1 to entry: This definition precludes the compositing of dissimilar materials, e.g. compositing textiles and paint coatings is not permitted.

4 Principle

The prepared test portion is digested in highly acidic conditions at high temperature using a hot plate digestion, a hot block digestion technique, or a microwave digestion system. Hot acid digestion destroys

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the material matrix allowing the elements of interest to be solubilised and quantified by a suitable analytical instrument (see Clause 9, detection limits of the instrumental method).

5 Reagents and apparatus

5.1 Reagents

Only reagents of recognized analytical grade or equivalent shall be used. The concentration of the analyte or interfering substances in the reagents and water shall be negligible compared to the lowest concentration to be determined.

"Trace metal" grade or equivalent reagents shall be used for the calibration standards used for the final instrumentation quantification stage.

5.1.1 Nitric acid, concentrated, 1,40 g/ml, 65 % (*v/v*), "analytical" grade.

5.1.2 Nitric acid, 10 % (*v/v*): Add 100 ml concentrated nitric acid (5.1.1) to 500 ml water (5.1.4). Dilute to 1 000 ml with water (5.1.4).

5.1.3 Hydrochloric acid, concentrated, 1,19 g/ml, 37 % (*v/v*), "analytical" grade.

5.1.4 Water, of at least grade 3 purity, in accordance with ISO 3696.

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5.1.5 Hydrogen peroxide, 30 % (*v/v*).

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NOTE Hydrogen peroxide which is not stabilized must be stored at cold (4 °C or less) temperatures.

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5.1.6 Methylene chloride, "analytical" grade.

5.1.7 Acetone/ethanol solution, 1:1 mixture of absolute ethanol and acetone ("analytical" grades).

5.2 Apparatus

All glassware shall be soaked in 10 % (*v/v*) nitric acid (5.1.2) for at least 2 h and then rinsed in deionised water before use.

5.2.1 Microwave digestion system

Microwave sample preparation system equipped with a sample holder and high-pressure microwave digestion vessels (5.2.2, high pressure microwave digestion vessel).

NOTE 1 Some newer models of microwave digestion systems do not utilise high-pressure digestion vessels and these systems are considered as a suitable alternative provided they give an equivalent performance.

NOTE 2 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 (see A.3, precautions relating to the use of microwave digestion).

5.2.2 High pressure microwave digestion vessel

Closed-top vessel specifically designed for microwave digestion, of suitable capacity. It is recommended to use a vessel capable of withstanding a temperature of at least 225 °C and an internal pressure of at least 3 000 kPa. The liner of the vessel shall be PTFE (polytetrafluoroethylene)/TFM [*tris*-(α-trifluoromethyl-β,β-difluorovinyl)-1,3,5-enzenetricarboxylate], or PTFE/PFA (perfluoroalkoxyethylene) or another

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chemically inert material. Vessels shall also be equipped with a safety relief valve or disc that will prevent vessel rupture or ejection of the vessel cap.

NOTE 1 The inner liners shall be inspected regularly to check for any chemical or physical degradation.

NOTE 2 Internal pressures in excess of 3 000 kPa can occur with some samples, e.g. crayons, and so a suitable pressure-rated vessel, e.g. 5 000 kPa, should be used in these cases.

5.2.3 Scalpel, or other suitable scraping or cutting tools.

5.2.4 Laboratory grinding mill.

5.2.5 Rotary grinder, preferably with carbide burr grinders.

5.2.6 Centrifuge, capable of centrifuging at (5 000 ± 500) g[1)], with compatible tubes.

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

5.2.8 Polypropylene or PFTE microfilters, pore size 0,45 µm.

5.2.9 Volumetric flasks, 25 ml or 100 ml capacity with stopper.

5.2.10 Pipettes, such as 1 ml, 5 ml, 10 ml, 20 ml, etc.

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5.2.11 Beakers, various capacities including 25 ml, 50 ml, 100 ml, etc.

5.2.12 Electric hot plate, suitable for operation at surface temperatures up to at least 140 °C.

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NOTE Provided that the hot plate is capable of handling the extra heating required, use of a 12 mm to 25 mm thick heat-resistant glass plate placed on the hot plate can help reduce the presence of hot spots common to electric hot plates.

5.2.13 Filter paper and funnel.

5.2.14 Hot block digester, heated metal block with variable temperature settings up to at least 140 °C (optionally can have programmable settings and temperature ramps) with compatible digestion vessels of suitable capacity.

6 Selection and composition of test portions

See A.1.2 (practical considerations in deciding whether to composite test portions).

6.1 Selection of test portions

Test portions shall be taken from accessible parts (see ISO 8124-1) of the laboratory sample in accordance with Clause 7 (preparation of test portions). When appropriate, the laboratory sample shall be subjected to relevant tests in accordance with ISO 8124-1, before the accessibility is considered. Identical materials in the laboratory sample can be combined and treated as a single test portion, but the use of additional laboratory samples is not permitted. If it is not possible to obtain at least 10 mg, no further testing shall be conducted and this shall be reported under Clause 11 c) (test report).

It is recommended that the test portion mass be in the region of 100 mg where sufficient material is available.

1) g=9,806 65 m/s

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6.2 Compositing of test portions

Up to three test portions can be combined to form a composite test portion provided that the required *detection limit* can still be achieved (see A.1.2, practical considerations in deciding whether to composite test portions) and the combined materials are similar in nature.

The compositing of dissimilar materials is not permitted, e.g. compositing textiles and paint coatings. When calculating the concentration of a target element in a material, it is assumed that all of that element found in the digested sample originated from any one of the composited materials. Using this assumption and the masses of the individual materials, the total concentration of the target element is calculated for each individual material in the composite test portion.

7 Preparation of test portions

Materials from the laboratory sample are selected for testing in accordance with Clause 6 (selection and composition of test portion) and removed using cutting tools such as scalpels, razor blades, scissors, and grinding and milling tools as described in the subclauses below. If a grinding apparatus [such as a mill (5.2.4) or rotary grinding tool (5.2.5) with disposable grinding bits] is used, then any contaminated parts shall be thoroughly cleaned or disposed of between uses to prevent cross-contamination. Ensure that the device itself cannot contaminate the material being prepared.

In 7.1 to 7.9, collect sufficient material to obtain a test portion of between 10 mg and 100 mg. In cases where less than 10 mg of material is available (see 6.1, selection of test portions) no further testing is required and this is reported under Clause 11 c) (test report).

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Digest the prepared test portion according to the procedures described in 8.1 (microwave digestion) or 8.2 (hot plate and hot block digestion of test portion).

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7.1 Coatings of paint, varnish, lacquer, printing ink, polymer and similar coatings

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Remove each different coating from the laboratory sample by scraping down to the base material, taking care to avoid the inclusion of the base material. Where lithographic coatings (dot printing) are present, it is impractical to separate the individual colours and so remove these coatings in such a way that a representative test portion is obtained.

For some coatings deposited on a non-polymeric base material, it is permissible to add a few drops of solvent, such as acetone/ethanol (5.1.7) mixture or methylene chloride (5.1.6), to soften the paint and aid in its removal from the base material.

In the first instance, acetone/ethanol (5.1.7) should be used. If this treatment is not effective in aiding removal, methylene chloride can be used under a fumes hood.

If a solvent treatment is used, ensure that all traces of solvent have been removed by evaporation prior to microwave digestion (see 8.1, microwave digestion). Divide removed coatings into small pieces having a maximum length in any direction of 2 mm in order to facilitate efficient digestion.

7.2 Polymeric and similar materials, including laminates, whether textile-reinforced or not, but excluding other textiles

Scrape-off, cut, or grind the clean, dry material into pieces having a maximum length in any dimension of 2 mm using a *scalpel* or other suitable scraping or cutting tool.

7.3 Paper, paperboard and cardboard

See A.2.2 (paper, paperboard and cardboard).

Cut the material into pieces with a maximum length in any dimension of 2 mm using a suitable cutting tool.

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