INTERNATIONAL STANDARD

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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

Reference number
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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.

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*:

— *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*

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.

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:

— 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;

— 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*

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

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

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

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.

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

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

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

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.

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.

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

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).

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).

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

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.

If the paper or paperboard to be tested is coated with paint, varnish, lacquer, printing ink, adhesive, or similar coating, test portions of the coating shall not be taken separately. In such cases, take test portions from the material so that they also include representative parts of the coated area.

Material that is printed, where the ink has become part of the base material, is prepared as though they are unprinted.

7.4 Natural or synthetic textiles

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

If the sample is not of a uniform material or colour, where possible, obtain a test portion from each different material or colour present in a mass greater than 100 mg. Materials or colours present in amounts between 10 mg and 100 mg shall form part of the test portion obtained from the main material.

Test portions taken from patterned textiles shall be representative of the whole material.

7.5 Other materials, whether mass-coloured or not

Cut, scrape, or grind the material into pieces having a maximum length in any dimension of 2 mm using a suitable cutting tool.

7.6 Materials intended to leave a trace

Obtain test portions from each different material in the laboratory sample, in the form that it appears in the laboratory sample, ensuring that the material is cut into pieces having a maximum length in any dimension of 2 mm.

7.7 Pliable modelling materials, including modelling clays, and gels

Obtain test portions from each different material or colour of material in the form that it appears in the laboratory sample, i.e. without allowing the material to dry out. Cut the material into pieces having a maximum length in any dimension of 2 mm.

7.8 Paints, including finger paints, varnishes, lacquers, and similar materials, in solid or liquid form

7.8.1 Materials in solid form

Using a suitable tool, grind, crush, or cut the sample into particles having maximum length in any dimension of 2 mm.

Finger paint supplied in the form of powder shall be diluted with water (5.1.4) in accordance with the manufacturer's instructions and then prepared according to 7.8.2 (materials in liquid form).

7.8.2 Materials in liquid form

Ensure that all settled material has been incorporated into the sample by scraping and long-term mechanical shaking. Immediately prior to sampling, ensure the liquid is homogenised by stirring or shaking for 5 min. 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 portion) no further testing is required and this is reported under Clause 11 c) (test report). Digest the prepared test portion according to 8.1 (microwave digestion) or 8.2 (hot place and hot block digestion of test portion).

If the liquid is intended to solidify or dry during intended use, it shall be coated onto a clean glass plate and dried to constant weight before taking test portions as described in 7.1 (coatings of paint, varnish, lacquer, printing ink, polymer and similar coatings).

Report under Clause 11 g) (test report) whether the material was tested in the dry or liquid state.

7.9 Metallic materials whether or not partly coated

Where practical, obtain the test portion from an uncoated part of the metallic material. If the metal part is partially coated, remove any coatings (including electroplated coatings) that can be scraped off. Electroplating that cannot be removed by scraping shall be tested together with the metallic base material.

NOTE Metallic materials that are completely coated so that no metal is accessible, as defined in ISO 8124-1, are not tested. However, this approach differs in legal texts in certain regions of the world. For example, the United States Consumer Product Safety Improvement Act of 2008 deems that surface coatings do not render the base material inaccessible. Furthermore, ISO 8124-3 only requires the testing of metallic components that are small parts, i.e. fit wholly within the test cylinder shown in ISO 8124-1, Figure 17.

Using suitable cutting and grinding tools, 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 portion) no further testing is required and this is reported under Clause 11 c) (test report).

8 Digestion of test portions and instrumental analysis

WARNING — Hydrogen peroxide can be used in hot plate and block digestions; but if used in microwave digestions, great care must be taken in order to avoid potential build-up of pressure during the heating cycle which could cause accidents and consequential loss of sample

The steps in 8.1 (microwave digestion) and 8.2 (hot plate and hot block digestion of test portion) are carried out under a fume hood or in the microwave digestion system (5.2.1).

Subclause 8.1 (microwave digestion) or 8.2 (hot plate and hot block digestion of test portion) describe how to digest the test portion using either aqua regia [3 parts hydrochloric acid (5.1.3) to 1 part nitric acid (5.1.1)] or reverse aqua regia [1 part hydrochloric acid (5.1.3) to 3 parts nitric acid (5.1.1)] followed by dilution and quantification using the instrumental technique of inductively coupled plasma – mass spectroscopy (ICP-MS) or inductively coupled plasma - atomic emission spectroscopy (ICP-AES). Other instrumental techniques can be used provided that they fulfil the criteria relating to detection limits set out in Table 1.

Aqua regia and reverse aqua regia are best prepared *in situ* in the presence of the test portion. The order of mixing the hydrochloric acid and nitric acid is not critical but should be the same for consistency.

Sample blanks shall be run with each batch of test portions digested according to 8.1 (microwave digestion) or 8.2 (hot plate and hot block digestion of test portion).

8.1 Microwave digestion

The microwave digestion system (5.2.1) nor the conditions to be used are specified in this part of ISO 8124. This allows the test laboratory to use any appropriate microwave system and digestion conditions. However, the test laboratory should determine the optimum digestion conditions based upon the equipment manufacturer's recommendations and the laboratory's own experiences with digestion of various material types. It is essential that complete digestion is achieved. In the event that the safety relief valve or system activates during the digestion, the analysis must be repeated using a new test portion and a suitably pressure rated vessel.

8.1.1 If the instrumental analysis technique is ICP-MS

Weigh accurately (to the nearest 0,1 mg) between 10 mg and 100 mg of the test portion prepared in Clause 7 (preparation of test portions) into a microwave digestion vessel (5.2.2) then carefully add 1,5 ml of concentrated hydrochloric acid (5.1.3) followed by 4,5 ml of concentrated nitric acid (5.1.1).

In order to reduce the potential for matrix interference, it is recommended to use the same grade reagents as the calibration standards.

Wait for the initial reaction to subside before sealing the vessel in accordance with the manufacturer's instructions.

8.1.2 If the instrumental analysis technique is ICP-AES

Replace the acids in 8.1.1 (if the instrumental analysis technique is ICP-MS) with the following mixture: 4,5 ml of concentrated hydrochloric acid (5.1.3) and 1,5 ml of concentrated nitric acid (5.1.1) and proceed as described in subclause 8.1.1.

8.1.3 Microwave digestion conditions

The microwave digestion system (5.2.1) shall be loaded with the sealed digestion vessel (5.2.2) and digested according to the conditions and temperatures recommended by the equipment manufacturers, as optimised by the test laboratory.

8.1.4 Cooling and dilution

Following completion of the digestion program, allow the vessels to cool for at least 5 min before transferring to a fumes hood for further cooling until the temperature of the sample is less than 40 °C (typically at least 1 h). Carefully open the vessel and check that the test portion has completely digested.

NOTE If the digestion is incomplete (e.g. evidence of original sample, solid lumps of charred test portion), the test should be repeated using a new test portion and alternative digestion conditions until a complete digestion has been achieved. In the event that a complete digestion cannot be achieved, an alternative method must be used (see A.4.1, incomplete digestion). Such methods are outside the scope of this part of ISO 8124 and their use shall be indicated in the final test report [see Clause 11 d), test report].

When the digestion is complete, quantitatively transfer the digestate with washings to a beaker and evaporate to about 1 ml on a hot plate. Allow to cool and then add about 4 ml to 5 ml water followed by 3 to 4 drops of hydrochloric acid (5.1.3). Filter (5.2.8 or 5.2.13) the digest solution into a 25 ml volumetric flask (5.2.9) or where limited sample was available, into a 10 ml volumetric flask. Dilute to the mark with water (5.1.4), seal the flask with a stopper and mix thoroughly. This diluted digest shall be subjected to instrumental quantification as soon as practically possible.

8.2 Hot plate and hot block digestion of test portion

WARNING — Fumes from nitric acid and hydrochloric acid are toxic; perform the following operations in a fume hood.

If arsenic or mercury is being quantified, the digestion technique shall use a closed vessel on the hot block digester (5.2.14) or in the microwave digestion system (5.2.1).

NOTE 1 Certain volatile elements such as arsenic and mercury are prone to be lost during hot plate and hot block digestion but laboratory testing has shown that mercury is not lost when using aqua regia acid mix.

NOTE 2 Aqua regia [three parts hydrochloric acid (5.1.3) to one part nitric acid (5.1.1)] or reverse aqua regia [one part hydrochloric acid (5.1.3) to three parts nitric acid (5.1.1)] are best prepared *in situ* in the presence of the test sample. The order of mixing the hydrochloric acid and nitric acid is not critical but should be the same for consistency.

8.2.1 If the instrumental analysis technique is ICP-AES

Weigh accurately (to the nearest 0,1 mg) between 10 mg and 100 mg of the test portion prepared in Clause 7 (preparation of test portions) into a clean 25 ml beaker (5.2.11) or hot block digestion vessel, then carefully add 4,5 ml of concentrated hydrochloric acid (5.1.3) followed by 1,5 ml concentrated nitric acid (5.1.1). Allow any reaction that may occur to subside, add 1 ml of hydrogen peroxide (5.1.5) (optional)

drop wise and cover with a watch glass if the reaction vessel is a beaker. Heat in a hot block (5.2.14) or on a hot plate (5.2.12) (surface temperature approximately 140 °C) until most of the acid has evaporated.

NOTE In the case of hot plate digestion, evaporate to a final volume of approximately 1 ml. The composition of this 1 ml digestate is predominantly concentrated nitric acid with reaction products from the sample. To eliminate the possibility of cross-contamination or sample loss, avoid boiling or evaporating to complete dryness.

If brown fumes are still observed after 1 h or particles are observed in the solution, continue the heating. Add a few millilitres of concentrated hydrochloric acid (5.1.3) and concentrated nitric acid (5.1.1) in the same ratio (3:1) as in 8.2.1 as necessary to prevent the sample from becoming dry. When the sample ceases to emit brown fumes, the digestion is complete. Remove the reaction vessel from the hot plate or hot block and allow to cool to room temperature.

NOTE In the event that a complete digestion cannot be achieved, an alternative method shall be used (see A.4.1, incomplete digestion).

8.2.1.2 Rinse the beaker and bottom of the watch glass with about 4 ml to 5 ml water (5.1.4) and then add 3 to 4 drops of concentrated hydrochloric acid. Filter (5.2.8 or 5.2.13) if necessary and then transfer the reaction mixture with washings into a 25 ml volumetric flask or where limited sample was available, into a 10 ml volumetric flask, make up to volume, seal the flask with a stopper and mix thoroughly. This diluted digest shall be subjected to instrumental quantification as soon as practically possible.

Where the digestate is made up to 25 ml volume, the diluted solution contains approximately 4 % (v/v) nitric acid (1 ml) with minor (<0.5 %) hydrochloric acid. Calibration standards used for instrumental analysis should be made with these levels of acids. Similar matching of acidity should be carried out if the digestate is made up to 10 ml.

Prior to instrumental quantification, remove any particulate in the solution by filtration (5.2.8 or 5.2.13), by centrifugation (5.2.6), or by allowing the solution to settle.

This diluted digest shall be subjected to instrumental quantification as soon as practically possible.

8.2.2 If the instrumental analysis technique is ICP-MS

Replace the acids in 8.2.1 with the following mixture: 1,5 ml of concentrated hydrochloric acid (5.1.3) and 4,5 ml of concentrated nitric acid (5.1.1) and proceed as described in subclause 8.2.1.

NOTE In order to reduce the potential for matrix interference, it is recommended to use the same grade reagents as the calibration standards.

9 Detection limits of the instrumental method

For the quantitative analysis of the diluted digests, the choice final instrumental analysis technique is left to the laboratory provided that it has detection limits not greater than those shown in Table 1. These values are 1/10th of maximum acceptable element migration from toy materials according to ISO 8124-3.

Table 1 — Maximum acceptable detection limit of an instrumental method

Values in milligrams per kilogram of toy material

Toy material	Element (mg/kg)							
	Sb	As	Ba	Cd	Cr	Pb	Hg	Se
Any toy material specified in 1.2 except modelling clay and finger paint	6	2	100	7	6	9	6	50
Modelling clay and finger paint	6	2	25	5	2	9	2	50

10 Expression of results

Calculate the concentration (mg/kg) of the target element by weight of material in the test portion according to the following formula:

$$C_T = \frac{C_E \times V_f \times D_f}{M} \quad (1)$$

where

C_T is the Concentration of target element in the toy material tested (mg/kg);

C_E is the Concentration of element measured by the instrument (mg/L);

V_f is the Volume of volumetric flask (ml);

D_f is the Dilution factor;

M is the Mass of the test portion or composite test portion (g).

Where a composite test portion was used, the results shall be calculated as described in A.1.2 (practical considerations in deciding whether to composite test portions).

11 Test report

The test report shall contain at least the following information:

a) type and identification of the product and/or material tested;

b) a reference to this part of ISO 8124 (ISO 8124-5:2015);

c) identification of materials that could not be tested due to insufficient mass (see 6.1, selection of test portion, and Clause 7, preparation of test portions);

d) the digestion method used (in cases where complete digestion cannot be achieved using the methods of this part of ISO 8124, the use of alternative methods can be applied, but their use shall be indicated in the test report together with a statement that they are outside the scope of this part of ISO 8124);

e) the test results calculated in accordance with Clause 10 (expression of results);

f) a statement on the estimated uncertainty of measurement when the uncertainty affects compliance to a specification limit or a customer's instruction so requires;

NOTE ISO/IEC Guide 98-3 or equivalent should be used to determine the uncertainty of measurement.

g) details of the selection and composition of test portions including whether composite test portions were used and in the case of liquid material, if it was tested in the liquid or dry state;

h) any departure, by agreement or otherwise, from the preparation and digestion procedures specified;

i) signature of responsible analyst or manager;

j) date of the test.

Annex A
(informative)

Background and rationale

A.1 Use and applicability

A.1.1 General

This part of ISO 8124 is intended to provide a means of determining whether a restricted element is present in a toy material and at what total concentration. This data may be used to decide whether further testing to ISO 8124-3:2010 is required. For example in the case that all elements of concern have a total concentration at or below the levels set out in ISO 8124-3:2010, Table 1 it would be impossible for the material to show a migration level in excess of the limits in Table 1.

NOTE This part of ISO 8124 has been developed only for the eight elements listed in Table 1. Test laboratories should undertake their own in-house validation of the methods detailed in this part of ISO 8124.

The total concentration data can also be used to determine whether other worldwide restrictions have been contravened. For example, in some countries there are limitations on the total concentration of lead in toy materials. Care must be taken to ensure that exempted materials and sample size limitations do not lead to incorrect conclusions.

As stated in 1.2 (Scope), this part of ISO 8124 is not applicable to glass, ceramic, and siliceous materials. Methods for the determination of total concentration of certain elements in these materials can be found in other standards such as CPSC-CH-E1002-08.1.

A.1.2 Practical considerations in deciding whether to composite test portions

Composite testing of test portions is an analytically valid process but care must be taken in order to avoid an incorrect interpretation of the analytical results. If very small sample masses are used in composite tests, errors in weighing can become significant. Therefore, wherever possible, it is recommended that the composite test portions masses are in the region of 100 mg. In cases where this is not practical, the mass of an individual test portion is not permitted to be less than 10 mg.

A sufficient amount of material has to be used for each material in the composite test portion, giving proper consideration for the weighing capabilities of the balance used, the detection limits following the dilution of the digested sample (see Table 1) and for the subsequent instrumental analysis.

The compositing of dissimilar materials is not permitted, e.g. compositing textiles and paint coatings. Only similar materials can be grouped together into a composite test portion.

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 just one of the composited materials. Using this assumption and the masses of the individual materials in the composite test portion, it is possible to calculate the total concentration of the target element in a single material.

In considering results from such a composite sample, it is imperative that a sufficient “safety factor” be applied to account for weighing inaccuracy and propagation of errors from each step in the analytical procedure to ensure non-conforming materials are correctly identified. It is recommended in a composite test portion of up to 3 individual test portions, any test portion having greater than 80 % of a regulatory limit, should be retested as a single test portion.

A worked example is shown below:

EXAMPLE Cadmium in polymeric materials