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Safety of toys —

Part 3:
Migration of certain elements

Sécurité des jouets —

Partie 3: Migration de certains éléments

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 8124-3 was prepared by Technical Committee ISO/TC 181, *Safety of toys*.

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

- *Part 1: Mechanical and physical properties*
- *Part 2: Flammability*
- *Part 3: Migration of certain elements*

Annexes A and B form an integral part of this part of ISO 8124. Annexes C, D and E are for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

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Introduction

The requirements of this part of ISO 8124 are based on the bioavailability of certain elements resulting from the use of toys and should not, as an objective, exceed the following levels per day:

- 1,4 µg for antimony[1];
- 0,1 µg for arsenic;
- 25,0 µg for barium;
- 0,6 µg for cadmium;
- 0,3 µg for chromium;
- 0,7 µg for lead;
- 0,5 µg for mercury;
- 5,0 µg for selenium.

For the interpretation of these values it has been necessary to identify an upper limit for the ingestion of toy material. Very limited data have been available for identifying this upper limit. As a working hypothesis, a summed average daily intake of the various toy materials has been gauged at the currently accepted value of 8 mg/d, being aware that in certain individual cases these values might be exceeded.

Combining the daily intake with the bioavailability values listed above, limits are obtained for various toxic elements in micrograms per gram of toy material (milligrams per kilogram) and are detailed in table 1. The values obtained have been adjusted to minimize children's exposure to toxic elements in toys and to ensure analytical feasibility, taking into account limits achievable under current manufacturing conditions (see annex D).

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[1] This level differs from the level of 0,2 µg given in EN 71-3:1994.

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Safety of toys —

Part 3:
Migration of certain elements

1 Scope

1.1 This part of ISO 8124 specifies maximum acceptable levels and methods of sampling and extraction prior to analysis for the migration of the elements antimony, arsenic, barium, cadmium, chromium, lead, mercury and selenium from toy materials and from parts of toys, except materials not accessible (see ISO 8124-1).

1.2 Maximum acceptable levels are specified for the migration of the elements listed in 1.1 from the following toy materials:

- coatings of paints, varnishes, lacquers, printing inks, polymers and similar coatings (see 8.1);
- polymeric and similar materials, including laminates, whether textile-reinforced or not, but excluding other textiles (see 8.2);
- paper and paper board, up to a maximum mass per unit area of 400 g/m^2 (see 8.3);
- natural or synthetic textiles (see 8.4);
- glass/ceramic/metallic materials, excepting lead solder when used for electrical connections (see 8.5);
- other materials, whether mass-coloured or not (e.g. wood, fibreboard, hardboard, bone and leather) (see 8.6);
- materials intended to leave a trace (e.g. the graphite materials in pencils and liquid ink in pens) (see 8.7);
- pliable modelling materials, including modelling clays, and gels (see 8.8);
- paints to be used as such in the toy, including finger paints, varnishes, lacquers, glazing powders and similar materials in solid or liquid form (see 8.9).

1.3 For the purposes of this part of ISO 8124, the following criteria are considered appropriate in the categorization of toys which can be sucked, licked or swallowed:

- all intended food/oral contact toys, cosmetic toys and writing instruments categorized as toys;
- toys intended for children up to six years of age, i.e. all accessible parts and components where there is a probability that those parts or components may come into contact with the mouth (see annex D).

Toys and parts of toys which, due to their accessibility, function, mass, size or other characteristics, obviously exclude any hazard due to sucking, licking or swallowing, bearing in mind the normal and foreseeable behaviour of children, are not covered by this part of ISO 8124.

1.4 Packaging materials are not included unless they are part of the toy or have intended play value (see annex D).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 8124. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 8124 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 8124-1:—[1], *Safety of toys — Part 1: Mechanical and physical properties.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

3 Definitions

For the purposes of this part of ISO 8124 the following definitions apply.

3.1 base material: Material upon which coatings may be formed or deposited.

3.2 coating: All layers of material formed or deposited on the base material 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, and which can be removed by scraping with a sharp blade.

3.3 detection limit of a method: Three times the standard deviation of the result obtained in the blank test using that method.

3.4 mass-coloured materials: Materials, such as wood, leather and other porous substances, which have absorbed colouring matter without formation of a coating.

3.5 scraping: Mechanical process for removal of coatings down to the base material.

3.6 toy material: All accessible materials present in a toy.

4 Maximum acceptable levels

4.1 Specific requirements

Toys and parts of toys as specified in clause 1 are deemed to meet the requirements of this part of ISO 8124 when the adjusted value of migration of elements from them comply with the maximum limits given in table 1 when tested in accordance with clauses 7, 8 and 9 (see annex D).

4.2 Interpretation of results

Due to the precision of the methods specified in this part of ISO 8124, an adjusted analytical result is required to take into consideration the results of interlaboratory trials. The analytical results obtained in accordance with clauses 7, 8 and 9 shall be adjusted by subtracting the analytical correction in table 2 to obtain an adjusted analytical result.

Materials are deemed to comply with the requirements of this part of ISO 8124, if the adjusted analytical result for the migrated element is less than or equal to the value given in table 1.

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[1] To be published.

Table 1 — Maximum acceptable element migration from toy materials

Values in milligrams per kilogram toy material

Toy material	Element							
	Sb	As	Ba	Cd	Cr	Pb	Hg	Se
Any toy material given in clause 1, except modelling clay and finger paint	60	25	1 000	75	60	90	60	500
Modelling clay and finger paint	60	25	250	50	25	90	25	500

Table 2 — Analytical correction

Element	Sb	As	Ba	Cd	Cr	Pb	Hg	Se
Analytical correction (%)	60	60	30	30	30	30	50	60

EXAMPLE:

An analytical result for lead of 120 mg/kg was obtained. The necessary analytical correction taken from table 2 is 30 %. Therefore the adjusted analytical result is

$$120 - \frac{120 \times 30}{100} = 120 - 36$$

$$= 84 \text{ mg/kg.}$$

This is deemed as complying with the requirements of this part of ISO 8124 (maximum acceptable migration of lead as given in table 1 is 90 mg/kg).

5 Principle

Soluble elements are extracted from toy materials under conditions which simulate the material remaining in contact with stomach acid for a period of time after swallowing. The concentrations of the soluble elements are then determined quantitatively by unspecified analytical methods with specified detection limits.

6 Reagents and apparatus

NOTE — No recommendation is made for the reagents, materials, and apparatus necessary for carrying out elemental analyses within the detection limits specified in clause 9.

6.1 Reagents

During the analyses, use only reagents of recognized analytical grade (see annex D).

6.1.1 Hydrochloric acid solution, $c(\text{HCl}) = (0{,}07 \pm 0{,}005)$ mol/l.

6.1.2 Hydrochloric acid solution, $c(\text{HCl}) = (0{,}14 \pm 0{,}010)$ mol/l.

6.1.3 Hydrochloric acid solution, $c(\mathrm{HCl})$ = approximately 1 mol/l.

6.1.4 Hydrochloric acid solution, $c(\mathrm{HCl})$ = approximately 2 mol/l.

6.1.5 Hydrochloric acid solution, $c(\mathrm{HCl})$ = approximately 6 mol/l.

6.1.6 1,1,1-trichloroethane, containing a maximum of 10 mg/kg of hydrochloric acid when tested in accordance with annex A, or other suitable solvent (see annex D).

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

6.2 Apparatus

Normal laboratory apparatus and

6.2.1 Plain-weave wire-cloth stainless steel metal sieve, of nominal aperture 0,5 mm and tolerances as indicated in table B.1.

6.2.2 Means of measuring pH with an accuracy of ± 0,2 pH units. Cross-contamination shall be prevented (see annex D).

6.2.3 Membrane filter, of pore size 0,45 µm.

6.2.4 Centrifuge, capable of centrifuging at $(5\,000 \pm 500)g$[1] (see annex D).

6.2.5 Means to agitate the mixture at a temperature of (37 ± 2) °C.

6.2.6 Series of containers, of gross volume between 1,6 times and 5,0 times that of the volume of hydrochloric acid extractant (see annex D).

7 Selection of test portions

A laboratory sample for testing shall consist of a toy either in the form in which it is marketed, or in the form in which it is intended to be marketed. Test portions shall be taken from accessible parts (see ISO 8124-1) of a single toy sample. When appropriate, the toy shall be subjected to relevant tests in accordance with ISO 8124-1, before the accessibility is considered. Identical materials in the toy may be combined and treated as a single test portion, but additional toy samples shall not be used. Test portions may be composed of more than one material or colour only if physical separation, e.g. dot printing, patterned textiles or mass limitation reasons, precludes the formation of discrete specimens. (See annex D.)

NOTE — The requirement does not preclude the taking of reference portions from toy materials in a different form provided that they are representative of the relevant material specified above and the substrate upon which they are deposited. (See annex D.)

Test portions of less than 10 mg of material shall not be tested.

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[1] $g = 9{,}806\,65\ \mathrm{m/s^2}$

8 Preparation and extraction of test portions

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

8.1.1 Test portion preparation

Remove the coating from the laboratory sample by scraping (3.5) at room temperature and comminute it at a temperature not exceeding ambient. Collect enough coating to obtain a test portion of preferably not less than 100 mg which will pass through a metal sieve of aperture 0,5 mm (6.2.1).

If only between 10 mg and 100 mg of comminuted uniform coating is available, extract this in accordance with 8.1.2 and calculate the quantity of the appropriate elements as if a test portion of 100 mg had been used. Report the mass of the test portion under 10 e).

In the case of coatings that by their nature cannot be comminuted (e.g. elastic/plastic paint), remove a test portion of coating from the laboratory sample without comminuting.

8.1.2 Extraction procedure

Using a container of appropriate size (6.2.6), mix the test portion prepared in 8.1.1 with 50 times its mass of an aqueous HCl solution at (37 ± 2) °C of c(HCl) 0,07 mol/l (6.1.1). [Where the test portion has only a mass of between 10 mg and 100 mg, mix the test portion with 5,0 ml of this solution (6.1.1) at (37 ± 2) °C.]

Shake for 1 min. Check the acidity of the mixture. If the pH is greater than 1,5, add dropwise, while shaking the mixture, an aqueous solution of c(HCl) approximately 2 mol/l (6.1.4) until the pH of the mixture is between 1,0 and 1,5.

Protect the mixture from light. Agitate the mixture continuously at (37 ± 2) °C (6.2.5) for 1 h and then allow to stand for 1 h at (37 ± 2) °C.

Without delay, efficiently separate the solids from the solution, firstly by filtration using a membrane filter (6.2.3) and, if necessary, by centrifuging at up to 5 000g (6.2.4). Carry out the separation as rapidly as possible after completion of the standing time. If centrifuging is used, it shall take no longer than 10 min and shall be reported under 10 e).

If the resulting solutions are to be stored for more than one working day prior to elemental analysis, stabilize them by addition of hydrochloric acid so that the concentration of the stored solution is approximately c(HCl) = 1 mol/l. Report such stabilization under 10 e).

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

8.2.1 Test portion preparation

Obtain a test portion of preferably not less than 100 mg of the polymeric or similar materials, whilst avoiding heating of the materials, according to the following procedure.

Cut out test portions from those areas having the thinnest material cross-section in order to ensure a surface area of the test pieces as large as possible in proportion to their mass. Each piece shall in the uncompressed condition have no dimension greater than 6 mm.

If the laboratory sample is not of a uniform material, obtain a test portion from each different material present in a mass greater than 10 mg. Where there is only between 10 mg and 100 mg of uniform material, report the mass of the test portion under 10 e) and calculate the quantity of the appropriate elements as if a test portion of 100 mg had been used.

8.2.2 Extraction procedure

Follow the extraction procedure in 8.1.2 using the test portions prepared in accordance with 8.2.1.

8.3 Paper and paper board

8.3.1 Test portion preparation (see annex D)

Obtain a test portion of preferably not less than 100 mg of the paper or paper board.

If the laboratory sample is not of a uniform material, obtain a test portion from each different material present in a mass of not less than 100 mg. Where there is only between 10 mg and 100 mg of uniform material, report the mass of the test portion under 10 e) and calculate the quantity of the appropriate elements as if a test portion of 100 mg had been used.

If the paper or paper board 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 and report this under 10 e). Extract test portions so obtained in accordance with 8.3.2. (See annex D.)

8.3.2 Extraction procedure

Macerate the test portion prepared in 8.3.1 with 25 times its mass of water (6.1.7) at (37 ± 2) °C so that the resulting mixture is homogeneous. Quantitatively transfer the mixture to the appropriate-sized container (6.2.6). Add to the mixture a mass of aqueous solution of $c(\text{HCl}) = 0{,}14$ mol/l (6.1.2) at (37 ± 2) °C which has 25 times the mass of the test portion.

Shake for 1 min. Check the acidity of the mixture. If the pH is greater than 1,5, add dropwise, while shaking the mixture, an aqueous solution of $c(\text{HCl})$ approximately 2 mol/l (6.1.4) until the pH of the mixture is between 1,0 and 1,5.

Protect the mixture from light. Agitate the mixture continuously at (37 ± 2) °C (see 6.2.5) for 1 h and then allow to stand for 1 h at (37 ± 2) °C.

Without delay, efficiently separate the solids from the solution, firstly by filtration using a membrane filter (6.2.3) and, if necessary, by centrifuging at up to 5 000*g* (see 6.2.4). Carry out the separation as rapidly as possible after completion of the standing time. If centrifuging is used, it shall take no longer than 10 min and shall be reported under 10 e).

If the resulting solutions are to be stored for more than one working day prior to elemental analysis, stabilize them by addition of hydrochloric acid so that the concentration of the stored solution is approximately $c(\text{HCl}) = 1$ mol/l. Report such stabilization under 10 e).

8.4 Natural or synthetic textiles

8.4.1 Test portion preparation

Obtain a test portion of preferably not less than 100 mg by cutting the textile material into pieces which in the uncompressed condition have no dimension greater than 6 mm. (See annex D.)

If the sample is not of a uniform material or colour, 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.

Samples taken from patterned textiles shall be representative of the whole material. (See annex D.)

8.4.2 Extraction procedure

Follow the extraction procedure in 8.1.2 using the test portions prepared in accordance with 8.4.1.

8.5 Glass/ceramic/metallic materials

8.5.1 Test portion preparation

Toys and toy components shall be first subjected to the small parts test in accordance with ISO 8124-1. If the toy or component fits entirely within the small parts cylinder and contains accessible glass, ceramic or metallic materials, then the toy or component shall be extracted in accordance with 8.5.2 after removal of any coating in accordance with 8.1.1. (See annex D.)

NOTE — Toys and toy components that have no accessible glass, ceramic or metallic materials do not require extraction in accordance with 8.5.2. (See annex D.)

8.5.2 Extraction procedure

Place the toy or toy component in a 50-ml glass cylinder with a nominal height of 60 mm and diameter of 40 mm.

NOTE — This type of container will take all components/toys that fit inside the small parts cylinder defined in ISO 8124-1.

Add a sufficient volume of an aqueous solution of $c(\text{HCl}) = 0{,}07$ mol/l (6.1.1) at (37 ± 2) °C to just cover the toy or component. Cover the container, protect the contents from light and allow the contents to stand for 2 h at (37 ± 2) °C.

Without delay, efficiently separate the solids from the solution, firstly by decantation followed by filtration using a membrane filter (6.2.3) and, if necessary, by centrifuging at up to 5 000*g* (6.2.4). Carry out the separation as rapidly as possible after completion of the standing time. If centrifuging is used, it shall take no longer than 10 min and shall be reported under 10 e).

If the resulting solutions are to be stored for more than one working day prior to elemental analysis, stabilize them by addition of hydrochloric acid so that the concentration of the stored solution is approximately $c(\text{HCl}) = 1$ mol/l. Report such stabilization under 10 e).

8.6 Other materials, whether mass-coloured or not (see annex D)

8.6.1 Test portion preparation

Obtain a test portion of preferably not less than 100 mg of the material in accordance with 8.2.1, 8.3.1, 8.4.1 or 8.5.1, as appropriate.

If the laboratory sample is not of uniform material, a test portion shall be obtained from each different material present in a mass greater than 10 mg. Where there is only between 10 mg and 100 mg of uniform material, report the mass of the test portion under 10 e) and calculate the quantity of the appropriate elements as if a test portion of 100 mg had been used.

If the material to be tested is coated with paint, varnish, lacquer, printing ink or similar coating, follow the procedure in 8.1.1.

8.6.2 Extraction procedures

Extract the materials in accordance with 8.2.2, 8.3.2, 8.4.2 or 8.5.2, as appropriate. Report the method used under 10 e).

8.7 Materials intended to leave a trace

8.7.1 Test portion preparation for materials in solid form

Obtain a test portion of preferably not less than 100 mg by cutting the material into pieces which in the uncompressed condition have no dimension greater than 6 mm.

A test portion shall be obtained from each different material intended to leave a trace, present in the laboratory sample in mass greater than 10 mg. Where there is only between 10 mg and 100 mg of material, report the mass of the test portion under 10 e) and calculate the quantity of the appropriate elements as if a test portion of 100 mg had been used.

If the material contains any grease, oil, wax or similar material, enclose the test portion in hardened filter paper and remove these ingredients with 1,1,1-trichloroethane or other suitable solvent (6.1.6) by extraction before treatment of the test portion as described in 8.7.4. Take analytical measures to ensure that removal of the ingredients referred to is quantitative. Report the solvent used under 10 e).

8.7.2 Test portion preparation for materials in liquid form

Obtain a test portion of preferably not less than 100 mg of the material from the laboratory sample. The use of an appropriate solvent to facilitate the obtaining of a test portion is permitted.

A test portion shall be obtained from each different material intended to leave a trace, present in the laboratory sample in mass greater than 10 mg. Where there is only between 10 mg and 100 mg of material, report the mass of the test portion under 10 e) and calculate the quantity of the appropriate elements as if a test portion of 100 mg had been used.

If the material is intended to solidify in normal use and contains grease, oil, wax or similar material, allow the test portion to solidify under normal-use conditions and enclose the resulting material in hardened filter paper. Remove the grease, oil, wax or similar material with 1,1,1-trichloroethane or other suitable solvent (6.1.6) by extraction before treatment of the test portion as described in 8.7.4. Take analytical measures to ensure that removal of the ingredients referred to is quantitative. Report the solvent used under 10 e).

8.7.3 Extraction procedure for samples not containing grease, oil, wax or similar material

Using a container of appropriate size (6.2.6), mix the test portion prepared in accordance with 8.7.1 or 8.7.2 with 50 times its mass of an aqueous HCl solution at (37 ± 2) °C of $c(\mathrm{HCl}) = 0{,}07$ mol/l (6.1.1). For a test portion of mass between 10 mg and 100 mg, mix the test portion with 5,0 ml of this solution at (37 ± 2) °C.

Shake for 1 min. Check the acidity of the mixture. If the test portion contains large quantities of alkaline materials, generally in the form of calcium carbonate, adjust the pH to between 1,0 and 1,5 with hydrochloric acid [c(HCl) approximately 6 mol/l (6.1.5)] in order to avoid overdilution. Report the amount of hydrochloric acid used to adjust pH in relation to the total amount of solution under 10 e).

If only a small quantity of alkaline material is present and the pH of the mixture is greater than 1,5, add dropwise, while shaking the mixture, an aqueous solution of c(HCl) approximately 2 mol/l (6.1.4) until the pH is between 1,0 and 1,5.

Protect the mixture from light. Agitate the mixture continuously at (37 ± 2) °C (6.2.5) for 1 h and then allow to stand for 1 h at (37 ± 2) °C prior to elemental analysis.

8.7.4 Extraction procedure for samples containing grease, oil, wax or similar material

With the test portion as prepared in 8.7.1 or 8.7.2 remaining in the hardened filter paper, macerate the test portion with a mass of water (6.1.7) at (37 ± 2) °C which has 25 times the mass of the original material so that the resulting mixture is homogeneous. Quantitatively transfer the mixture to a container of appropriate size (6.2.6). Add to the mixture a mass of aqueous solution of $c(\mathrm{HCl}) = 0{,}14$ mol/l (6.1.2) at (37 ± 2) °C which has 25 times the mass of the original test portion.

In the case of a test portion of original mass between 10 mg and 100 mg, macerate the test portion with 2,5 ml or water (6.1.7). Quantitatively transfer the mixture to the appropriate-sized container (6.2.6). Add 2,5 ml aqueous solution of $c(\mathrm{HCl}) = 0{,}14$ mol/l (6.1.2) at (37 ± 2) °C to the mixture.

Shake for 1 min. Check the acidity of the mixture. If the test portion contains large quantities of alkaline materials, generally in the form of calcium carbonate, adjust the pH to between 1,0 and 1,5 with hydrochloric acid of c(HCl) approximately 6 mol/l (6.1.5) in order to avoid overdilution. Report the amount of hydrochloric acid used to adjust pH in relation to the total amount of solution under 10 e).

If only a small quantity of alkaline material is present and the pH of the mixture is greater than 1,5, add dropwise, while shaking the mixture, an aqueous solution of c(HCl) approximately 2 mol/l (6.1.4) until the pH is between 1,0 and 1,5.

Protect the mixture from light. Agitate the mixture continuously at (37 ± 2) °C (6.2.5) for 1 h and then allow to stand for 1 h at (37 ± 2) °C.

NOTE — The volume of HCl solution of c(HCl) = 0,07 mol/l (see 8.7.3) or c(HCl) = 0,14 mol/l, as the case may be, is calculated on the mass of the test portion prior to dewaxing.

Without delay, efficiently separate the solids from the solution, firstly by filtration using a membrane filter (6.2.3) and, if necessary, by centrifuging at up to 5 000g (6.2.4). Carry out the separation as rapidly as possible after completion of the standing time. If centrifuging is used, it shall take no longer than 10 min and shall be reported under 10 e).

If the resulting solutions are to be stored for more than one working day prior to elemental analysis, stabilize them by addition of hydrochloric acid so that the concentration of the stored solution is approximately c(HCl) = 1 mol/l. Report such stabilization under 10 e).

8.8 Pliable modelling materials, including modelling clays, and gels

8.8.1 Test portion preparation

Obtain a test portion of not less than 100 mg of the material from the laboratory sample; obtain a test portion from each different material in the laboratory sample.

If the material contains grease, oil, wax or similar material, enclose the test portion in hardened filter paper and remove these ingredients with 1,1,1-trichloroethane or other suitable solvent (6.1.6) by extraction before treatment of the test portion as described in 8.8.3. Take analytical measures to ensure that removal of the ingredients referred to is quantitative. Report solvent used under 10 e).

8.8.2 Extraction procedure for samples not containing grease, oil, wax or similar material

Using a container of appropriate size (6.2.6) mix the test portion prepared in accordance with 8.8.1, after breaking up of clay or doughy materials if appropriate, with 50 times its mass of an aqueous solution at (37 ± 2) °C of c(HCl) = 0,07 mol/l (6.1.1).

Shake the mixture for 1 min. Check the acidity of the mixture. If the test portion contains large quantities of alkaline materials, generally in the form of calcium carbonate, adjust the pH to between 1,0 and 1,5 with hydrochloric acid of c(HCl) approximately 6 mol/l (6.1.5) in order to avoid overdilution. Report the amount of hydrochloric acid used in relation to the total amount of solution under 10 e).

If only a small quantity of alkaline material is present and the pH of the mixture is greater than 1,5, add dropwise, while shaking the mixture, an aqueous solution of c(HCl) approximately 2,0 mol/l (6.1.4) until the pH is between 1,0 and 1,5.

Protect the mixture from light. Agitate the mixture continuously at (37 ± 2) °C (6.2.5) for 1 h and then allow to stand for 1 h at (37 ± 2) °C.

8.8.3 Extraction procedure for samples containing grease, oil, wax or similar material

With the test portion as prepared in 8.8.1 remaining in the hardened filter paper, macerate the test portion with a mass of water (6.1.7) at (37 ± 2) °C which has 25 times the mass of the original material so that the resulting mixture is homogeneous. Quantitatively transfer the mixture to a container of appropriate size (6.2.6). Add to the mixture a mass of aqueous solution of c(HCl) = 0,14 mol/l (6.1.2) at (37 ± 2) °C which has 25 times the mass of the original test portion.

Shake for 1 min. Check the acidity of the mixture. If the test portion contains large quantities of alkaline materials, generally in the form of calcium carbonate, adjust the pH to between 1,0 and 1,5 with an aqueous solution of c(HCl) approximately 6 mol/l (6.1.5) in order to avoid overdilution. Report the amount of hydrochloric acid used in relation to the total amount of solution under 10 e).

If only a small quantity of alkaline material is present and the pH of the mixture is greater than 1,5, add dropwise, while shaking the mixture, an aqueous solution of c(HCl) approximately 2,0 mol/l (6.1.4) until the pH is between 1,0 and 1,5.

Protect the mixture from light. Agitate the mixture continuously at (37 ± 2) °C (6.2.5) for 1 h and then allow to stand for 1 h at (37 ± 2) °C.

NOTE — The volume of the HCl solution of c(HCl) = 0,07 mol/l (see 8.8.2) or c(HCl) = 0,14 mol/l, as the case may be, is calculated on the mass of the test portion prior to dewaxing.

Without delay, efficiently separate the solids from the solution, firstly by filtration using a membrane filter (6.2.3) and, if necessary, by centrifuging at up to 5 000g (6.2.4). Carry out the separation as rapidly as possible after completion of the standing time. If centrifuging is used, it shall take no longer than 10 min and shall be reported under 10 e).

If the resulting solutions are to be stored for more than one working day prior to elemental analysis, stabilize them by addition of hydrochloric acid so that the concentration of the stored solution is approximately c(HCl) = 1 mol/l. Report such stabilization under 10 e).

8.9 Paints, including finger paints, varnishes, lacquers, glazing powders and similar materials in solid or liquid form

8.9.1 Test portion preparation for materials in solid form

Obtain a test portion of preferably not less than 100 mg of the material, if appropriate by scraping off the material or by cutting into pieces which in the uncompressed condition have no dimension greater than 6 mm.

A test portion shall be obtained from each different material present in the laboratory sample in mass greater than 10 mg. Where there is only between 10 mg and 100 mg of material, report the mass of the test portion under 10 e) and calculate the quantity of the appropriate elements as if a test portion of 100 mg had been used.

If the material contains any grease, oil, wax or similar material, enclose the test portion in hardened filter paper and remove these ingredients with 1,1,1-trichloroethane or other suitable solvent (6.1.6) by extraction before treatment of the test portion as described in 8.9.4. Take analytical measures to ensure that removal of the ingredients referred to is quantitative. Report the solvent used under 10 e).

If the test portion is removed by scraping, comminute the test portion so that the material will pass through a metal sieve with an aperture of 0,5 mm (6.2.1).

8.9.2 Test portion preparation for materials in liquid form

Obtain a test portion of preferably not less than 100 mg of the material from the laboratory sample. The use of an appropriate solvent to facilitate the obtaining of a test portion is permitted.

A test portion shall be obtained from each different material present in the laboratory sample and forming a mass greater than 10 mg. Where there is only between 10 mg and 100 mg of material, report the mass of the test portion under 10 e) and calculate the quantity of the appropriate elements as if a test portion of 100 mg had been used.

If the material is intended to solidify in normal use and contains grease, oil, wax or similar material, allow the test portion to solidify under normal-use conditions and enclose the resulting material in hardened filter paper. Remove the grease, oil, wax or similar material with 1,1,1-trichloroethane or other suitable solvent (6.1.6) by extraction before treatment of the test portion as described in 8.9.4. Take analytical measures to ensure that removal of the ingredients referred to is quantitative. Report the solvent used under 10 e).

8.9.3 Extraction procedure for samples not containing grease, oil, wax or similar material

Follow the extraction procedure in 8.7.3 using the test portions prepared in accordance with 8.9.1 or 8.9.2.

8.9.4 Extraction procedure for samples containing grease, oil, wax or similar material

With the test portion as prepared in 8.9.1 or 8.9.2 remaining in the filter paper, macerate the test portion with a mass of water (6.1.7) at (37 ± 2) °C which has 25 times the mass of the original material so that the resulting mixture is homogeneous. Quantitatively transfer the mixture to a container of appropriate size (6.2.6). Add to the mixture a mass of aqueous solution of $c(\text{HCl}) = 0{,}14$ mol/l (6.1.2) at (37 ± 2) °C which has 25 times the mass of the original test portion.

Shake for 1 min. Check the acidity of the mixture. If the test portion contains large quantities of alkaline materials, generally in the form of calcium carbonate, adjust the pH to between 1,0 and 1,5 with an aqueous solution of $c(\text{HCl})$ approximately 6 mol/l (6.1.5) in order to avoid overdilution. Report the amount of hydrochloric acid used in relation to the total amount of solution under 10 e).

If only a small quantity of alkaline material is present and the pH of the mixture is greater than 1,5, add dropwise, while shaking the mixture, an aqueous solution of $c(\text{HCl})$ approximately 2 mol/l (6.1.4) until the pH is between 1,0 and 1,5.

Protect the mixture from light. Agitate the mixture continuously at (37 ± 2) °C (6.2.5) for 1 h and then allow to stand for 1 h at (37 ± 2) °C.

NOTE — The volume of the solution of $c(\text{HCl}) = 0{,}07$ mol/l (see 8.7.3) or $c(\text{HCl}) = 0{,}14$ mol/l, as the case may be, is calculated on the mass of the test portion prior to dewaxing.

Without delay, efficiently separate the solids from the solution, firstly by filtration using a membrane filter (6.2.3) and, if necessary, by centrifuging at up to 5 000*g* (6.2.4). Carry out the separation as rapidly as possible after completion of the standing time. If centrifuging is used, it shall take no longer than 10 min and shall be reported under 10 e).

If the resulting solutions are to be stored for more than one working day prior to elemental analysis, stabilize them by addition of hydrochloric acid so that the concentration of the stored solution is approximately $c(\text{HCl}) = 1$ mol/l. Report such stabilization under 10 e).

9 Detection limits of quantitative elemental analysis

For the quantitative analysis of the extracts of toys for the migrated elements listed in clause 1, methods having a detection limit of a maximum of 1/10 of the values to be determined (see 4.1, table 1) shall be applied. The detection limit (of the analytical method) is deemed to be three times the standard deviation of the blank value as measured by the laboratory carrying out the analysis of the toy materials.

Laboratories using methods deviating from this requirement shall report the detection limit under 10 c).

10 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 8124-3:1997);

c) the technique used for determining the quantity of each migrated element, and the detection limit if different from the requirements in clause 9;

d) the adjusted results (see 4.2) of the quantitative elemental analysis, expressed as milligrams migrated element per kilogram toy material, stating that the result is related to the element in solution;

e) details of the procedure used, in accordance with requirements in clause 8;

f) any departure, by agreement or otherwise, from the preparation and extraction procedures specified;

g) date of the test.

Annex A
(normative)

Determination of acidity of 1,1,1-trichloroethane

A.1 Reagents

A.1.1 Standard sodium hydroxide solution, $c(\text{NaOH}) = (0{,}1 \pm 0{,}005)$ mol/l.

A.1.2 Phenolphthalein indicator, 0,5 g in 100 ml of 95 % (*V/V*) (volume fraction 95 %) ethanol, turned faintly pink by the addition of the minimum quantity of dilute aqueous hydrochloric acid or dilute aqueous sodium hydroxide.

A.1.3 Water, neutralized by the addition of dilute aqueous sodium hydroxide using a few drops of the phenolphthalein indicator.

A.2 Test procedure

Place 100 ml of neutralized water (A.1.3) in a 250 ml flask, add 100 ml of 1,1,1-trichloroethane test sample, stopper the flask and shake vigorously. Allow the layers to separate, add 0,5 cm^3 of phenolphthalein indicator and titrate with 0,1 mol/l aqueous sodium hydroxide (A.1.1) using a microburette until the upper layer turns a faint pink colour.

A.3 Calculation

The acidity, A, of 1,1,1-trichloroethane, expressed as micrograms of hydrochloric acid per gram of trichloroethane, is calculated from the following equation:

$$A = \frac{36{,}5}{\rho} V$$

where

V is the volume of 0,1 mol/l sodium hydroxide, expressed in millilitres;

ρ is the relative density of the test sample of 1,1,1-trichloroethane.

Annex B
(normative)

Sieve requirements

Table B.1 — Sieve dimensions and tolerances

Dimensions in millimetres

Nominal aperture size	**Nominal wire diameter in test sieve**	**Tolerances**		
		Maximum deviation for size of an individual aperture	Tolerance for average aperture	Intermediate deviation (no more than 6 % of the apertures shall exceed the nominal aperture size plus this number)
0,500	0,315	+ 0,090	± 0,018	+ 0,054

Annex C
(informative)

Selection of procedure

The diagram in figure C.1 is a guideline for the choice of procedure to be used for the various toy materials.

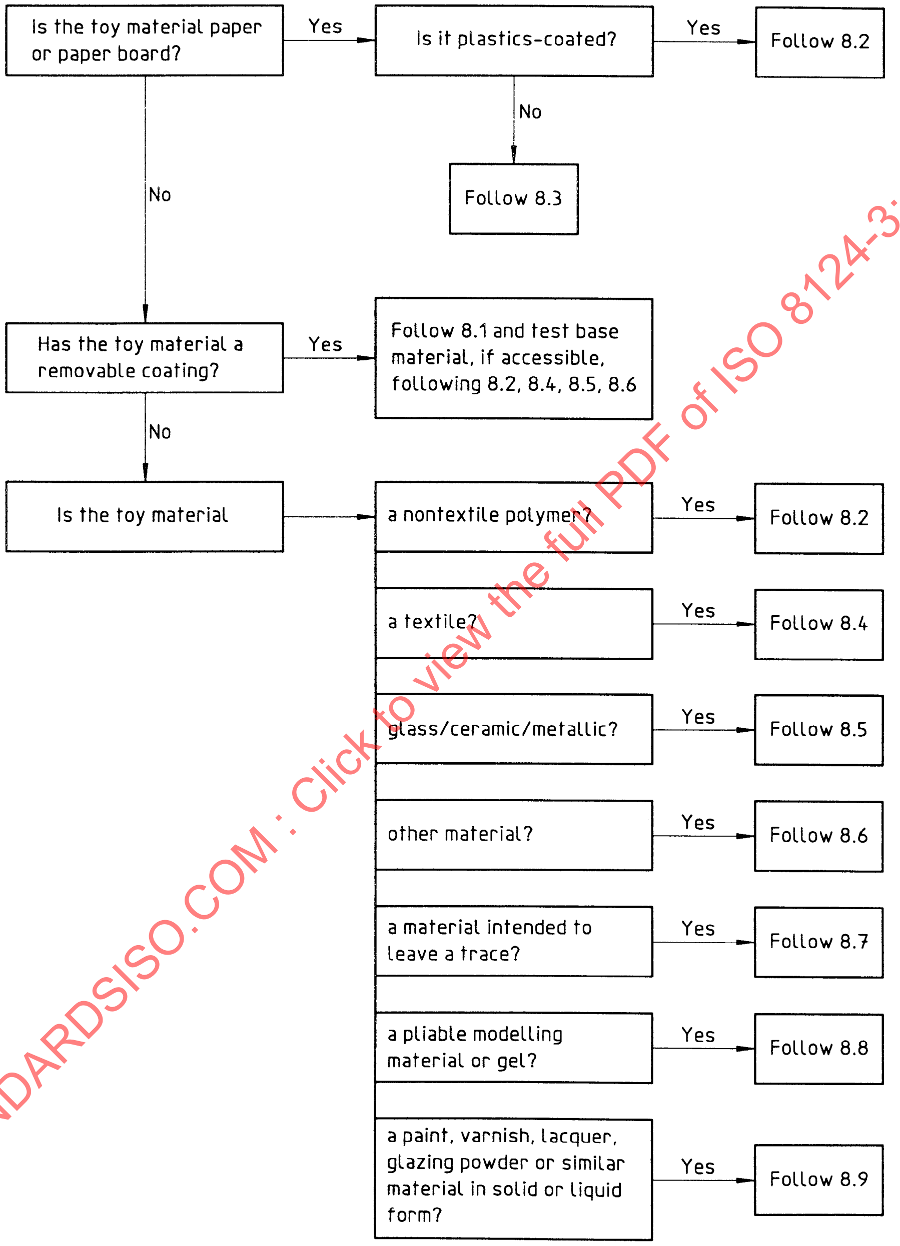

Figure C.1 — Guidelines for choosing the procedure for preparation and extraction of test portions

Annex D
(informative)

Background and rationale

D.1 Introduction

The way bioavailability is defined in the European Council Directive 88/378/EEC of May 1988 concerning the safety of toys (published in the Official Journal of the EC No. L 187 of 16 July 1988) led to the methods in this part of ISO 8124 addressing the migration levels of soluble elements from a toy material.

The approach using total migrated element determinations was discarded for the following reasons.

a) The above-mentioned Directive indicates bioavailability limits, and there has been no link to date between the availability of an element in a toy material with respect to extraction with simulated gastric solutions and the total content of that element in the material;

b) Some compounds, such as barium sulfate, can be included in products at a 2 % level to render them radio-opaque. This use of barium would either have to be excluded from toys or a separate requirement made.

c) Cadmium compounds can be used as stabilizers in plastics such as polyvinylchloride (PVC). These compounds under normal conditions are insoluble in simulated gastric solution. Their use therefore, if total elements were determined, would have to be accounted for in the requirements of this part of ISO 8124. A case can be made for selenium, which can be present as a constituent of insoluble pigments, etc. (See also D.4).

D.2 Scope

D.2.1 Subclause 1.3

Subclause 1.3 is intended to indicate an approach to the decision of what toys, or toy components, are excluded from this part of ISO 8124 because of characteristics that render them unlikely to present a risk of injury from the ingestion of materials containing toxic elements.

This was considered a logical approach for a number of reasons, including:

— as children get older, the tendency for them to place toys/toy materials in their mouths is reduced and therefore the risk of the ingestion of toxic elements is reduced;

— the larger the toy or the less accessible the material, the lower the risk of ingestion of toxic elements.

It was therefore considered that all toys which might be placed in the mouth, or close to the mouth would be tested, e.g. pencils.

Toys intended for children over the age of six years were considered not to pose a significant risk of injury through the ingestion of toxic elements.

D.2.2 Packaging (subclause 1.4)

"Unless part of the toy" in 1.4 is intended to mean, for example, boxes containing jigsaw puzzles or packaging on which the instructions are included in the case of games, etc., but taking into account the second listed item of 1.3 restricting the requirements to toys intended for children up to six years of age. It is not intended to address, for example, blister packs containing simple instructions.

D.3 Specific requirements (see 4.1)

The maximum acceptable level of soluble barium has been raised from 500 mg/kg to 1 000 mg/kg for the following reasons:

- the use of barium sulfate in toys has led to levels of soluble barium in the acidic extraction solution [c(HCl) = (0,07 ± 0,005) mol/l at 37 °C] of between 400 mg/kg and 600 mg/kg as expressed on the mass of toy material taken. This level is such that, due to the statistical uncertainty of the determination, a PASS or FAIL could not be indicated;
- the formation of non-bioavailable colloidal barium sulfate crystals in the filtrate resulted in levels of apparently soluble barium which exceeded 500 mg/kg because of problems with filtration;
- in addition, the previous maximum level of migration of barium of 500 mg/kg from toy materials was not consistent with 25,0 µg/d bioavailability and the 8 mg/d of toy material intake; 25,0 µg corresponds to a migration limit of 3,125 mg/kg. It is understood that the 500 mg/kg limit was consciously selected despite the "theoretical" 3,125 mg/kg value. The effect of the 500 mg/kg limit was to reduce the bioavailability from the advised 25 µg to 4 µg. It should be noted that the 25,0 µg value has been reduced from an initial 50,0 µg, not for toxicological reasons, but according to the Commission of the European Communities, "to reduce the avoidable input on body burden".

D.4 Statistical uncertainty of the test procedure and interpretation of results (see 4.2)

Most chemical test methods are designed to measure the *total* amount of a substance in a material. This makes it easier to obtain an accurate result with close statistical agreement between laboratories because there is an absolute or true value.

Because of the way bioavailability is defined in the European Council Directive 88/378/EEC, the methods in this part of ISO 8124 lead to measurement of the migration of *soluble* element from a toy material. With this approach, the analytical result is dependent upon the specified conditions of test and there is no absolute or true value. Consequently, it is more difficult to obtain close statistical agreement between laboratories when performing such migration tests.

This is illustrated by the statistical information in EN 71-3:1988 taken from a 1987 European interlaboratory trial involving 17 laboratories. Results on an identical material varied by at least 30 % and up to 50 % between laboratories, depending on the instrumental technique used to measure the soluble element concentration of the filtrate. Moreover, these figures would be approximately three times higher if adjusted to a 95 % confidence level.

This degree of statistical uncertainty creates problems for manufacturers and enforcement authorities if test results are near the maximum limits allowed in this part of ISO 8124. Then it is not statistically possible to pass or fail a toy and leads to inconsistency in the interpretation of results.

There is no direct relationship between the actual total element content of a toy material and the soluble migration of that element under standard test conditions. Therefore, measuring the total element content and converting the result to give a soluble element value is not an answer to this problem. Setting maximum total element limits is a possibility, but requires an amendment to the European Council Directive 88/378/EEC (see also D.1).

Since 1988, the procedure for extraction from paint coatings on toys has been thoroughly investigated to determine which parameters significantly effect the results. The most critical parameters are the shape, size and mass of the paint particles produced by removal of the paint and its subsequent comminution. Other less critical parameters include the method of shaking, the temperature, and the type and porosity of filter paper.

As a result, a procedure for scraping and comminution to collect a paint test portion of particle size 300 µm to 500 µm was specified as a proposed revision to the procedure. A European interlaboratory trial was conducted in 1993 with 29 participating laboratories to compare the amended procedure with that in EN 71-3:1988.

The trial showed that results on an identical material can vary by at least 25 % and up to 80 % depending on the method of preparation of the test portions and the instrumental technique used to measure the soluble-element concentration of the filtrate.

Statistical agreement between laboratories showed improvement using a specified scraping procedure, but not when collecting a test portion of particle size 300 µm to 500 µm. However, any improvement was not significant enough to justify the proposed changes.

The trial confirmed that the use of different instrumental techniques contribute to the statistical uncertainty of the test procedure. It was also noted that laboratories need to check and calibrate their instruments on a regular basis to ensure accurate readings. Inductively Coupled Plasma (ICP) was more widely used by laboratories this time, and tended to show better agreement for most of the elements, particularly arsenic, antimony and selenium. However, it is not as sensitive as hydride-generation methods for low levels of the same elements.

A test procedure that produces results varying, at best, by 25 % between laboratories would normally be considered as technically unsuitable as a reference method. However, in reality, toys will either easily pass or fail this test, and only in relatively few cases will a result fall within the area of uncertainty. When this occurs, it is important that laboratories interpret the results in the same way.

It has been accepted that the test procedure cannot be improved without imposing time-consuming, costly, and in some cases, impractical burdens on laboratories with little benefit in terms of statistical agreement and safety. Therefore, the procedure allows laboratories to use their preferred techniques for scraping the paint off toys, collecting the portion that passes through a 500 µm sieve and determining the soluble-element concentration of the filtrate.

To achieve consistent interpretation of results, a correction factor for each element and applicable to all instrumental techniques has been introduced into this part of ISO 8124. These are taken from the precision data in EN 71-3:1988 and are used when an analytical result equals or exceeds the maximum limit. The analytical result is adjusted as described in 4.2, using the relevant correction factor. This way of interpreting the results is perfectly adequate as a screening test to differentiate between safe and unsafe toys as well as to ensure the safety of children.

In future, it is recommended that laboratories check and compare their performance of the test procedure by using reference materials and participating in a proficiency scheme.

D.5 Reagents (see 6.1)

D.5.1 1,1,1-trichloroethane (see 6.1.6)

This subclause limits the maximum level of acidity that 1,1,1-trichloroethane is permitted to contain. As is well known, this solvent breaks down in sunlight to form hydrochloric acid.

Under the Montreal Protocol, 1,1,1-trichloroethane will be phased out for general use. This part of ISO 8124 indicates that other suitable solvents may be used, but these will need to be evaluated to ensure that they are as efficient at wax/grease extraction as is 1,1,1-trichloroethane.

D.6 Apparatus (see 6.2)

D.6.1 Plain-weave wire cloth stainless steel sieve (see 6.2.1)

See D.4.

D.6.2 Means of measuring pH (see 6.2.2)

The measurement of pH is not restricted to the use of a pH-meter.