4.2 Interpretation of results

See D.4.

Due to the precision of the methods specified in this document, an adjusted analytical result is required to take into consideration the results of interlaboratory trials. The analytical results obtained in accordance with $\frac{\text{Clauses 8}}{\text{Clauses 10}}$, $\frac{9}{\text{Clauses 10}}$ and $\frac{10}{\text{Clauses 10}}$ shall be adjusted by subtracting the analytical correction in $\frac{\text{Table 2}}{\text{Clauses 10}}$ to obtain an adjusted analytical result.

Materials are deemed to conform with the requirements of this document if the adjusted analytical result for the migrated element is less than or equal to the value given in <u>Table 1</u>.

Table 1 — Maximum acceptable element migration from toy materials

Values in milligrams per kilogram of toy material

Toy material	Element								
	Sb	As	Ba	Cd	Cr	Pb	Hg	Se	
Any toy material given in <u>Clause 1</u> , except modelling clay and finger paint	60	25	1 000	75	60	90	60	500	
Modelling clay	60	25	250	50	25	90	25	500	
Finger paint	10	10	350	15	25	25	10	50	

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 as shown in Formula (1).

$$120 - \frac{120 \times 30}{100} = 120 - 36 \tag{1}$$

= 84 mg/kg.

This is deemed to be conforming with the requirements of this document (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 that 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 inductively coupled plasma optical emission spectrometry (ICP-OES) or other specified analytical methods with specified MDL.

6 Reagents

NOTE No recommendation is made for the reagents and materials necessary for carrying out elemental analyses within the MDL specified in 10.2.

During the analyses, use only reagents of recognized analytical grade.

- **6.1** Hydrochloric acid solution, $c(HCl) = (0.07 \pm 0.005) \text{ mol/l}.$
- **6.2** Hydrochloric acid solution, $c(HCl) = (0.14 \pm 0.010) \text{ mol/l}.$

- **6.3 Hydrochloric acid solution**, c(HCI) = approximately 1 mol/l.
- **6.4 Hydrochloric acid solution**, c(HCI) = approximately 2 mol/l.
- **6.5 Hydrochloric acid solution**, c(HCl) = approximately 6 mol/l.
- **6.6** General purpose reagent n-heptane, (C₇H₁₆), 99 %.
- **6.7 Water of at least grade 3 purity**, in accordance with ISO 3696.

7 Apparatus

NOTE No recommendation is made for the apparatus necessary for carrying out elemental analyses within the MDL specified in 10.2.

See D.5.

Normal laboratory apparatus and the following:

7.1 Plain-weave wire-cloth stainless steel metal sieve, of nominal aperture 0,5 mm and tolerances as indicated in Annex A, Table A.1.

7.2 Means of measuring pH to proper accuracy without cross-contamination

The accuracy of pH measurement shall be cautiously considered to make sure the pH value of the mixture is in the range 1,0-1,5 (as specified in Clause 9). For example, when a pH measurement with an accuracy of \pm 0,2 pH units is used, the range shall be changed to 1,2-1,3 after considering the uncertainty.

See D.5.1.

- 7.3 Membrane filter, of pore size 0,45 μm.
- **7.4 Centrifuge**, capable of centrifuging at $(5\ 000 \pm 500)\ g^{1)}$.

See D.5.2.

- **7.5 Means of agitating the mixture**, at a temperature of (37 ± 2) °C.
- **7.6 Series of containers**, of gross volume between $1.6 \times \text{and } 5.0 \times \text{that of the volume of hydrochloric acid extractant.}$

See D.5.3.

- **7.7 Soxhlet extractor**, according to ISO 8124-6:2018, Figure C.1.
- **7.8** Solvent extractor, according to ISO 8124-6:2018, Figure C.2.
- **7.9 High retention filter paper**, for example quantitative slow flow rate filter paper.

8 Selection of test portions

See D.6.

¹⁾ $1 g = 9,806 65 \text{ m/s}^2$.

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 the accessible parts (see ISO 8124-1) of a single toy sample. 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, such as dot printing, patterned textiles or mass limitation reasons, precludes the formation of discrete specimens.

NOTE The requirement does not preclude the taking of test portions from materials used to manufacture the toy, provided they are representative of the final toy.

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

9 Preparation and extraction of test portions

NOTE A guideline for the choice of procedure to be used for the various toy materials is provided in Annex B.

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

9.1.1 Test portion preparation

Remove the coating from the laboratory sample by scraping (see 3.7) 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 (7.1).

If only between 10 mg and 100 mg of comminuted uniform coating is available, extract this in accordance with 9.1.2 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 in accordance with Clause 11 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.

9.1.2 Extraction procedure

Using a container of appropriate size (7.6), mix the test portion prepared in 9.1.1 with $50 \times$ its mass of an aqueous HCl solution at (37 ± 2) °C of c(HCl) 0,07 mol/l (6.1). [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) at (37 ± 2) °C.]

Shake for 1 min. Check the acidity of the mixture (7.2). If the pH is greater than 1,5, add dropwise, while shaking the mixture, an aqueous solution of c(HCl) approximately 2 mol/l (6.4) 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 (7.5) 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 (7.3) and, if necessary, by centrifuging at up to 5 000 g (7.4). 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 in accordance with Clause 11 e).

If the resulting solutions are to be stored for more than one working day prior to elemental analysis, stabilize them by adding hydrochloric acid so that the concentration of the stored solution is approximately $c(HCl) = 1 \text{ mol/l} (\underline{6.3})$. Report such stabilization in accordance with <u>Clause 11</u> e).

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

9.2.1 Test portion preparation

Obtain a test portion of preferably not less than 100 mg of the polymeric or similar material, while 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 of 10 mg or more. Where there is only between 10 mg and 100 mg of uniform material, report the mass of the test portion in accordance with <u>Clause 11</u> e) and calculate the quantity of the appropriate elements as if a test portion of 100 mg had been used.

9.2.2 Extraction procedure

Follow the extraction procedure in 9.1.2 using the test portions prepared in accordance with 9.2.1.

9.3 Paper and paperboard

9.3.1 Test portion preparation

See D.7.

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

If the laboratory sample is not of a uniform material, where possible, 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 in accordance with <u>Clause 11</u> e) and calculate the quantity of the appropriate elements as if a test portion of 100 mg had been used.

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 and report this in accordance with Clause 11 e). Extract test portions obtained in accordance with 9.3.2.

9.3.2 Extraction procedure

Macerate the test portion prepared in 9.3.1 with 25 × its mass of water (6.7) at (37 ± 2) °C so that the resulting mixture is homogeneous. Quantitatively transfer the mixture to the appropriate-sized container (7.6). Add to the mixture a mass of aqueous solution of c(HCl) = 0.14 mol/l (6.2) at (37 ± 2) °C which has 25 × the mass of the test portion.

Shake for 1 min. Check the acidity of the mixture (7.2). If the pH is greater than 1,5, add dropwise, while shaking the mixture, an aqueous solution of c(HCl) approximately 2 mol/l (6.4) 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 (7.5) 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 (7.3) and, if necessary, by centrifuging at up to $5\,000\,g$ (7.4). Carry out the separation as rapidly as possible after completion of the standing time. If centrifuging is used, it shall take no longer than $10\,\text{min}$ and shall be reported in accordance with Clause $11\,\text{e}$).

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If the resulting solutions are to be stored for more than one working day prior to elemental analysis, stabilize them by adding hydrochloric acid so that the concentration of the stored solution is approximately c(HCl) = 1 mol/l. Report such stabilization in accordance with Clause 11 e).

9.4 Natural, artificial or synthetic textiles

9.4.1 Test portion preparation

See D.8.

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

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.

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

9.4.2 Extraction procedure

Follow the extraction procedure in 9.1.2 using the test portions prepared in accordance with 9.4.1.

9.5 Glass/ceramic/metallic materials

9.5.1 Test portion preparation

See D.9.

Toys and toy components shall first be 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 (excepting lead solder when used for electrical connections), then the toy or component shall be extracted in accordance with 9.5.2 after removal of any coating in accordance with 9.1.1.

NOTE Toys and toy components that have no accessible glass, ceramic or metallic materials do not require extraction in accordance with <u>9.5.2</u>.

9.5.2 Extraction procedure

Place the weighed 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 measured volume of an aqueous solution of c(HCl) = 0.07 mol/l (6.1) 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 (7.3) and, if necessary, by centrifuging at up to 5 000 g (7.4). 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 in accordance with Clause 11 e).

If the resulting solutions are to be stored for more than one working day prior to elemental analysis, stabilize them by adding hydrochloric acid so that the concentration of the stored solution is approximately c(HCl) = 1 mol/l (6.3). Report such stabilization in accordance with Clause 11 e).

9.6 Other materials, whether mass-coloured or not (e.g. wood, fibreboard, bone and leather)

9.6.1 Test portion preparation

See D.10.

Obtain a test portion of preferably not less than 100 mg of the material in accordance with <u>9.2.1</u>, <u>9.3.1</u>, <u>9.4.1</u> or <u>9.5.1</u>, 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 of 10 mg or more. Where there is only between 10 mg and 100 mg of uniform material, report the mass of the test portion in accordance with <u>Clause 11</u> 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 a similar coating, follow the procedure in 9.1.1.

9.6.2 Extraction procedures

Extract the materials in accordance with 9.1.2, 9.3.2 or 9.5.2, as appropriate. Report the method used in accordance with Clause 11 e).

9.7 Materials intended to leave a trace

9.7.1 Test portion preparation for materials in solid form

9.7.1.1 General

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 a mass of 10 mg or more. Where there is only between 10 mg and 100 mg of material, report the mass of the test portion in accordance with <u>Clause 11</u> 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, these ingredients shall be removed before treatment of the test portion as described in 9.7.4. Two alternative dewaxing methods are described in 9.7.1.2 and 9.7.1.3 as Method A and Method B. Laboratories can select the most suitable dewaxing method at their discretion. Other methods may also be used but shall be validated to show that they are capable of completely removing non-polar ingredients from relevant toy materials.

9.7.1.2 Method A

Enclose the test portion in a high retention filter paper (7.9) and place into the thimble of a Soxhlet extractor (7.7). Add 50 ml of n-heptane (6.6) to the 100 ml boiling flask of the extractor and reflux for at least 30 min with no less than five reflux cycles. At the conclusion of the process, dry the folded filter paper containing the dewaxed test portion to ensure the removal of residual solvent. Take analytical measures to ensure that the removal of the ingredients referred to is quantitative. Report the solvent used in accordance with Clause 11 e).

The filter paper used shall be as small as possible without risking loss of the test portion during the dewaxing procedure.

NOTE The volume of n-heptane can be adjusted according to the Soxhlet extractor used.