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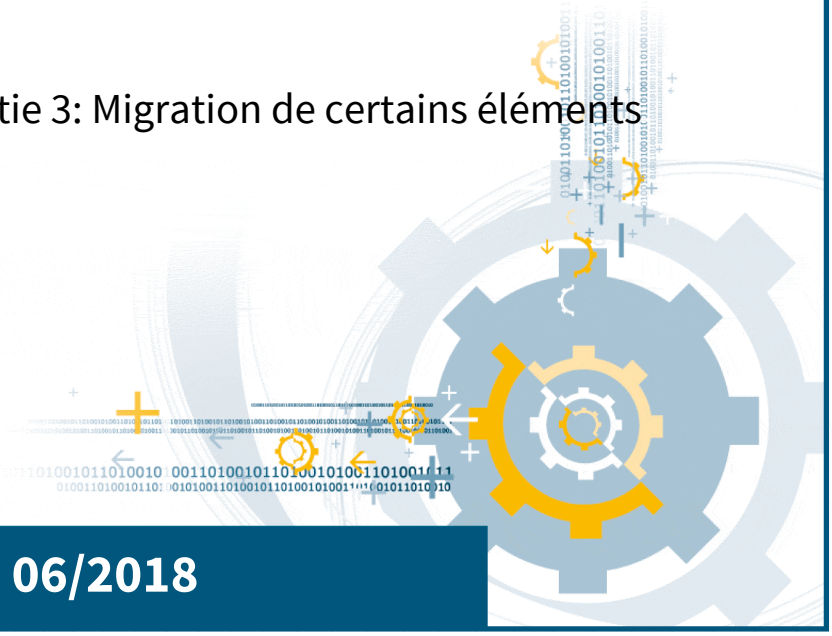
Institut luxembourgeois de la normalisation
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des produits et services

ILNAS-EN 71-3:2013+A3:2018

Safety of toys - Part 3: Migration of certain elements

Sicherheit von Spielzeug - Teil 3: Migration bestimmter Elemente

Sécurité des jouets - Partie 3: Migration de certains éléments



06/2018

National Foreword

This European Standard EN 71-3:2013+A3:2018 was adopted as Luxembourgish Standard ILNAS-EN 71-3:2013+A3:2018 in June 2018.

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 71-3:2013+A3

June 2018

ICS 97.200.50

Supersedes EN 71-3:2013+A2:2017

English Version

Safety of toys - Part 3: Migration of certain elements

Sécurité des jouets - Partie 3: Migration de certains éléments

Sicherheit von Spielzeug - Teil 3: Migration bestimmter Elemente

This European Standard was approved by CEN on 29 Mai 2013 and includes Amendment 1 approved by CEN on 18 August 2014, Amendment 2 approved by CEN on 18 April 2017 and Amendment 3 approved by CEN on 15 March 2018.

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Contents

Page

European foreword ... 5
Introduction ... 7
1 Scope ... 7
2 Normative references ... 7
3 Terms and definitions ... 8
4 Requirements ... 9
4.1 Toy material categories (see H.4) ... 9
4.2 Specific requirements ... 9
5 Principle ... 10
6 Reagents and apparatus ... 11
6.1 Reagents ... 11
6.2 Apparatus ... 11
7 Sampling and sample preparation ... 12
7.1 Selection of test portions ... 12
7.2 Standards preparation ... 12
7.3 Sample preparation ... 12
7.3.1 General ... 12
7.3.2 Category I: Dry, brittle, powder like or pliable and Category II: Liquid or sticky ... 12
7.3.3 Category III: Scraped-off ... 13
7.4 Migration procedure ... 14
7.4.1 General ... 14
7.4.2 Category I: Dry, brittle, powder like or pliable and Category II: Liquid or sticky ... 14
7.4.3 Category III: Scraped-off ... 15
8 Methods of analysis ... 17
9 Calculation of results ... 17
9.1 General elements ... 17
9.1.1 Calibration curve ... 17
9.1.2 Calculation of migration ... 18
9.1.3 Interpretation of results ... 18
9.2 Chromium (III) and Chromium (VI) ... 18
9.2.1 Calibration curve ... 18
9.2.2 Calculation of migration ... 18
9.2.3 Interpretation of results ... 19

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9.3 Organic tin 19
9.3.1 Calibration curves 19
9.3.2 Calculation of migration of single organic tin compound 19
9.3.3 Calculation of migration of organic tin 20
9.3.4 Interpretation of results 20
10 Test report 20
Annex A (informative) Significant technical changes between this European Standard and the previous version 21
Annex B (informative) Method performance 23
B.1 Method performance 23
B.2 Use of measurement uncertainty in compliance assessment 23
B.3 Information on round robin test 23
Annex C (normative) Sieve requirements 25
Annex D (informative) Preparation and analysis of test portion 26
Annex E (informative) Method of analysis for general elements 27
E.1 Principle 27
E.2 Working solutions 27
E.3 Procedure 29
E.4 Analysis 29
Annex F (informative) Methods of analysis for speciation of Chromium (III) and (VI) 32
F.1 Principle 32
F.2 Reagents 32
F.3 Apparatus 33
F.4 Procedure 34
F.5 Analysis 34
Annex G (informative) Method of analysis for organic tin (see H.10) 37
G.1 Principle 37
G.2 Terms and definitions 38
G.3 Reagents 38
G.4 Apparatus 41
G.5 Procedure 41
G.6 Analysis 42
Annex H (informative) Rationale 47
H.1 General 47
H.2 Mouthing behaviour of children (Scope) 47
H.3 Skin contact (Scope) 47
H.4 Toy categories (4.1) 48

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H.5 Test portions (7.1, 7.3.3.2, 7.3.3.3, 7.3.3.6) 48
H.6 Size of test pieces (7.3.3.2, 7.3.3.3, 7.3.3.4) 48
H.7 Textile threads 48
H.8 Chromium (VI) migration (7.4) 48
H.9 Filtration of migration solutions (7.4.2.1, 7.4.2.2, 7.4.3.1, 7.4.3.3, 7.4.3.5) 49
H.10 Organic tin (9.3) 49
H.11 pH value 49
H.12 De-waxing (see 7.3.2) 50
Annex I (informative) Conformity assessment 51
I.1 General 51
I.2 Chromium VI 51
Annex J (informative) List of other organic tin compounds 52
Annex ZA (informative) Relationship between this European Standard and the Essential Requirements of EU Directive 2009/48/EC aimed to be covered 53
Bibliography 54

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European foreword

This document (EN 71-3:2013+A3:2018) has been prepared by Technical Committee CEN/TC 52 "Safety of toys", the secretariat of which is held by DS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2018, and conflicting national standards shall be withdrawn at the latest by December 2018.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes A3 EN 71-3:2013+A2:2017 A3.

This document includes Amendment 1 approved by CEN on 2014-08-18.

This document includes Amendment 2 approved by CEN on 2017-04-18.

This document includes Amendment 3 approved by CEN on 2018-03-15.

The start and finish of text introduced or altered by amendments is indicated in the text by tags A1 A1, A2 A2 and A3 A3.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive 2009/48/EC.

For relationship with EU Directive 2009/48/EC, see informative Annex ZA, which is an integral part of this document.

The significant changes from the previous edition of this standard are detailed in Annex A.

EN 71, *Safety of toys*, consists of the following parts:

— *Part 1: Mechanical and physical properties*;

— *Part 2: Flammability*;

— *Part 3: Migration of certain elements*;

— *Part 4: Experimental sets for chemistry and related activities*;

— *Part 5: Chemical toys (sets) other than experimental sets*;

— *Part 7: Finger paints — Requirements and test methods*;

— *Part 8: Activity toys for domestic use*;

— *Part 9: Organic chemical compounds — Requirements*;

— *Part 10: Organic chemical compounds — Sample preparation and extraction*;

— *Part 11: Organic chemical compounds — Methods of analysis*;

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— *Part 12: N-Nitrosamines and N-nitrosatable substances*;

— *Part 13: Olfactory board games, gustative board games, cosmetic kits and gustative kits*;

— *Part 14: Trampolines for domestic use*.

NOTE 1 In addition to the above parts of EN 71, the following guidance documents have been published: CEN Technical Report CEN/TR 15071, *Safety of toys — National translations of warnings and instructions for use in* EN 71, CEN Technical Report CEN/TR 15371-1, Safety of toys - Interpretations - Part 1: Replies to requests for interpretation of EN 71-1, EN 71-2, EN 71-8 and EN 71-14 and CEN/TR 15371-2, Safety of toys - Interpretations - Part 2: Replies to requests for interpretation of the chemical standards in the EN 71-series.

NOTE 2 Words in *italics* are defined in Clause 3 (Terms and definitions).

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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Introduction

The Toy Safety Directive (2009/48/EC) [1] specifies maximum migration limits for three categories of *toy materials*. The limits for the migration of certain elements are expressed in milligram per kilogram *toy material* and are detailed in Table 2. The purpose of the limits is to minimize children's exposure to certain potentially toxic elements.

1 Scope

This European Standard specifies requirements and test methods for the migration of aluminium, antimony, arsenic, barium, boron, cadmium, chromium (III), chromium (VI), cobalt, copper, lead, manganese, mercury, nickel, selenium, strontium, tin, organic tin and zinc from *toy materials* and from parts of toys.

Packaging materials are not considered to be part of the toy unless they have intended play value.

NOTE 1 See guidance document of the European Commission guidance document no. 12 [2] on the application of the Directive on the safety of toys – packaging.

The standard contains requirements for the migration of certain elements from the following categories of *toy materials*:

— Category I: Dry, brittle, powder like or pliable materials;

— Category II: Liquid or sticky materials;

— Category III: Scraped-off materials.

The requirements of this standard do not apply to toys or parts of toys which, due to their accessibility, function, volume or mass, clearly exclude any hazard due to sucking, licking or swallowing or prolonged skin contact when the toy or part of toy is used as intended or in a foreseeable way, bearing in mind the behaviour of children.

NOTE 2 For the purposes of this standard, for the following toys and parts of toys the likelihood of sucking, licking or swallowing toys is considered significant (see H.2 and H.3):

— all toys intended to be put in the mouth or to the mouth, cosmetics toys and writing instruments categorized as toys can be considered to be sucked, licked or swallowed;

— all the accessible parts and components of toys intended for children up to 6 years of age can be considered to come into contact with the mouth. The likelihood of mouth contact with parts of toys intended for older children is not considered significant in most cases (see H.2).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 71-1:2011, *Safety of toys — Part 1: Mechanical and physical properties*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

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3 Terms and definitions

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

3.1
base material
material upon which *coatings* may be formed or deposited

3.2
coating
layer of material formed or deposited on a *base material* which can be removed by *scraping*

Note 1 to entry: *Coatings* can include paints, varnishes, lacquers, inks, polymeric *coatings* or other substances of a similar nature, whether they contain metallic particles or not, and irrespective of the manner of application.

3.3
limit of detection
minimum single result which, with a stated probability, can be distinguished from a suitable blank value; the result, c_L, is given by the formula:

$$c_L = \overline{x}_{bl} + k \times s_{bl}$$

where

〚A1〛 $\overline{x}_{bl}$ is the mean of the blank values,

s_{bl} is the standard deviation of the blank values and 〚A1〛

k is a numerical factor chosen according to the confidence level desired

Note 1 to entry: The use of $k = 3$ will usually suffice.

3.4
limit of quantification
lowest amount of analyte in a sample that can be quantitatively determined with a suitable precision and accuracy

Note 1 to entry: For the purpose of this standard, the limit of quantification is twice the limit of detection.

3.5
other material, whether mass coloured or not
material such as wood, leather or other porous substances which may absorb colouring matter without forming a *coating*

3.6
paper
sheet formed by irregularly intervened fibres with a mass per unit area of 400 g/m^2 or less

3.7
paper board
sheet formed by irregularly intervened fibres with a mass per unit area over 400 g/m^2

Note 1 to entry: The term *paperboard* also includes materials commonly referred to as card or cardboard with a mass per unit area over 400 g/m^2.

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3.8
scraping
mechanical removal of *coatings* down to but not including the *base material*

3.9
toy material
material present in toys and accessible as determined in accordance with EN 71-1:2011, 8.10

4 Requirements

4.1 Toy material categories (see H.4)

Table 1 shows the categories of common *toy materials*. Toys not listed in Table 1 shall be classified into one of the categories.

Table 1 — Cross-reference table for determining category

Toy Material	Category I	Category II	Category III
Coatings of paints, varnishes, lacquers, printing inks, polymers, foams and similar *coatings*			X
Polymeric and similar materials, including laminates, whether textile reinforced or not, but excluding other textiles			X
Paper and paper board			X
Textiles, whether natural or synthetic			X
Glass, ceramic, metallic materials			X
Other materials whether mass coloured or not (e.g. wood, fibre board, hard board, bone and leather)			X
Compressed paint tablets, materials intended to leave a trace or similar materials in solid form appearing as such in the toy (e.g. the cores of colouring pencils, chalk, crayons)	X		
Pliable modelling materials, including modelling clays and plaster [3]	X		
Liquid paints, including finger paints, varnishes, lacquers, liquid ink in pens and similar materials in liquid form appearing as such in the toy (e.g. slimes, bubble solution)		X	
Glue sticks		X	

4.2 Specific requirements

The migration of elements from *toy materials* categorized in accordance with 4.1 shall not exceed the migration limits given in Table 2 when tested in accordance with Clauses 7 and 8.

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Table 2 — Migration limits from toy materials

Element	Migration limit		
	Category I mg/kg	Category II mg/kg	Category III mg/kg
Aluminium	5 625	1 406	70 000
Antimony	45	11,3	560
Arsenic	3,8	0,9	47
Barium	1 500	375	18 750
Boron	1 200	300	15 000
Cadmium	1,3	0,3	17
Chromium (III)	37,5	9,4	460
Chromium (VI)	0,02	0,005	0,2
Cobalt	10,5	2,6	130
Copper	622,5	156	7 700
Lead	2,0	0,5	23
Manganese	1 200	300	15 000
Mercury	7,5	1,9	94
Nickel	75	18,8	930
Selenium	37,5	9,4	460
Strontium	4 500	1 125	56 000
Tin	15 000	3 750	180 000
Organic tin	0,9	0,2	12
Zinc	3 750	938	46 000

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5 Principle

Soluble elements are extracted from *toy materials* using conditions which simulate the material remaining in contact with gastric juices for a period of time after swallowing. The concentrations of the soluble elements are determined quantitatively by three different methods:

— method for determining general elements: Aluminium, Antimony, Arsenic, Barium, Boron, Cadmium, Chromium, Cobalt, Copper, Lead, Manganese, Mercury, Nickel, Selenium, Strontium, Tin and Zinc;

— method for determining Chromium (III) and Chromium (VI);

— method for determining organic tin.

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6 Reagents and apparatus

6.1 Reagents

All reagents used for analysis shall be of analytical grade or, if unavailable, technical grade reagents which have been determined to have acceptably low levels of impurity to allow the analysis to be performed.

6.1.1 Hydrochloric acid solution, c(HCl) = (0,07 ± 0,005) mol/l.

6.1.2 Hydrochloric acid solution, c(HCl) = (0,14 ± 0,010) mol/l.

6.1.3 Hydrochloric acid solution, c(HCl) = approximately 1 mol/l.

6.1.4 Hydrochloric acid solution, c(HCl) = approximately 2 mol/l.

6.1.5 Hydrochloric acid solution, c(HCl) = approximately 6 mol/l.

6.1.6 n-Heptane, (C_7H_{16}), 99 %.

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

6.2 Apparatus

Standard laboratory equipment and the following shall be used.

6.2.1 Plain weave wire cloth stainless steel sieve, of nominal aperture size 0,5 mm and tolerances as indicated in Table C.1.

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6.2.2 Equipment for measuring pH, calibrated and sufficiently accurate for the purposes of this European Standard. A1

6.2.3 Centrifuge, capable of centrifuging at high speed to separate the solids.

6.2.4 A means to agitate the mixture, at a temperature of (37 ± 2) °C.

An orbital or linear shaker, wrist action shaker, shaking water bath or magnetic stirrer can be used. It is important that the migration solution is in constant motion relative to the sample.

6.2.5 A selection of containers of gross volume, between 1,6 times and 5,0 times that of the volume of hydrochloric acid extract.

NOTE It is important to be aware the use of borosilicate glass ware may result in high blank values for Boron.

6.2.6 High retention filter-paper, ashless filter paper, particle retention in liquids 2,5 µm.

6.2.7 Membrane filters with pore sizes of 0,45 µm, 0,22 µm and 0,02 µm.

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7 Sampling and sample preparation

7.1 Selection of test portions

A laboratory sample for testing shall consist of one toy in the form in which it will be marketed. Test portions shall be taken from *toy materials* of the 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 to prepare larger test portions. Test portions are taken from each colour of each *toy material*. Test portions may be composed of more than one *toy material* or colour only when discrete specimens cannot be separated physically, e.g. dot printing, patterned textiles, etc.

NOTE This requirement does not preclude the preparation of test portions which represent the material and any *base material* upon which it is deposited.

If the total weight of available *toy material* is less than 10 mg test portions are disregarded (see H.5).

The above does not preclude the possibility of testing *toy materials* before they are used to manufacture a toy in order to prove compliance of the final toy. In this case, it shall be assessed that the manufacturing process does not influence the migration of elements from the *toy materials*.

7.2 Standards preparation

Prepare standard solutions covering a suitable working range appropriate to the required limits for each element for the three categories. Examples for ICP-MS and ICP-OES are included in Annex E.

7.3 Sample preparation

7.3.1 General

A blank solution shall be determined for any bias. The analytical results shall be corrected for any bias due, for example, to contaminants present in the filter paper (6.2.6) or the gastric simulant.

When there is a significant bias the method should be checked.

7.3.2 Category I: Dry, brittle, powder like or pliable and Category II: Liquid or sticky

If possible obtain a test portion of not less than 100 mg of the material from the laboratory sample.

If a test portion of 100 mg or more cannot be obtained, a test portion shall be obtained from each toy material present in the laboratory sample in a mass greater than 10 mg. If the weight of the test portion obtained is between 10 mg and 100 mg, that weight shall be reported (see Clause 10 h)). In this case the analytical results shall be calculated as though 100 mg of the test portion had been used (see H.5).

If the toy material contains any grease, oil, wax or similar material, material shall be de-waxed. De-waxing shall not be carried out on test portions being analysed for organic tin. Dry a high retention filter paper (see 6.2.6) for 4 h at 37 °C ± 4°C. Weigh the filter paper to the nearest 0,1 mg (Wfp1). The filter paper used should be as small as possible without risking loss of the test portion during the de-waxing procedure. Weigh the test portion, to the nearest 0,1 mg, onto the pre-dried and weighed filter paper. Calculate the mass of the test portion. Use this mass for the calculation of the results of the test. Fold the filter paper carefully to enclose the test portion without loss. Extract the test portion within the filter paper with boiling n-heptane (6.2.5) using suitable laboratory apparatus. The use of a de-waxing step shall be reported (see Clause 10 h)).

It has been shown that Soxhlet extraction with n-heptane for 6 h is usually sufficient to completely remove non-polar ingredients from waxy toy materials. Alternative methods should be validated to show that they are capable of completely removing the non-polar ingredients from relevant toy materials.

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After removal of non-polar ingredients, dry the folded filter paper containing the dewaxed test portion in an oven at (37 ± 2) °C for 4 h to ensure the removal of residual solvent. Weigh the dried filter paper parcel, to the nearest 0,1 mg (Wfp2). Use Wfp2 for the calculation under 7.4.2.1 on the volume of 0,07 M HCl.

7.3.3 Category III: Scraped-off

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

Remove the *coating* from the laboratory sample by mechanical means (usually *scraping*) at room temperature. If possible obtain a test portion of not less than 100 mg passing through a metal sieve of aperture 0,5 mm (see 6.2.1).

If the weight of the test portion obtained is between 10 mg and 100 mg, that weight shall be reported (see Clause 10 h)). In this case the analytical results shall be calculated as though 100 mg of the test portion had been used (see H.5). When possible, *coatings* on textiles are to be scraped off as a powder (and report this under Clause 10 h)). In case of a thick layer or one which is difficult to remove (e.g. pliable or plasticised layers), the *coating* can be cut off and tested as polymeric material (7.3.3.2).

7.3.3.2 Polymeric and similar materials including laminates and reinforced textiles, but excluding other textiles

If possible obtain a test portion of not less than 100 mg of the polymeric or similar material, according to the following directions.

Cut out test portions from the areas having the thinnest material cross section. Each test piece shall have at least one dimension of approximately 6 mm when possible (see H.6). The use of pre-prepared reference materials for visual size comparison is recommended.

If a test portion of 100 mg or more cannot be obtained, a test portion shall be obtained from each *toy material* present in the laboratory sample in a mass greater than 10 mg. If the weight of the test portion obtained is between 10 mg and 100 mg, that weight shall be reported (see Clause 10 h)). In this case the analytical results shall be calculated as though 100 mg of the test portion had been used (see H.5).

7.3.3.3 *Paper* and *paper board*

If possible obtain a test portion of not less than 100 mg of the *paper* or *paper board*. Each test piece shall have at least one dimension of approximately 6 mm when possible (see H.6). The use of pre-prepared reference materials for visual size comparison is recommended.

If a test portion of 100 mg or more cannot be obtained, a test portion shall be obtained from each *toy material* present in the laboratory sample in a mass greater than 10 mg. If the weight of the test portion obtained is between 10 mg and 100 mg, that weight shall be reported (see Clause 10 h)). In this case the analytical results shall be calculated as though 100 mg of the test portion had been used (see H.5).

If the *paper or paper board* to be tested has paint, varnish, lacquer, printing ink, adhesive or similar material applied to its surface, test portions of the *coating* shall not be taken separately. In such cases test portions shall be taken from the *toy material* so that they also include representative parts of the coated area.

7.3.3.4 Textiles, whether natural or synthetic (see H.7)

If possible obtain a test portion of not less than 100 mg of the textile material by cutting into test pieces.

Each test piece shall have at least one dimension of approximately 6 mm when possible (see H.6). The use of pre-prepared reference materials for visual size comparison is recommended.

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If a sample is not uniform in its material or colour, a test portion shall be obtained from each different material present in a mass greater than 100 mg. A test portion present in a mass between 10 mg and 100 mg shall not be tested separately but shall be tested together with the material to which it is attached. Test portions taken from patterned textiles shall be representative of the whole material.

7.3.3.5 Glass, ceramic and metallic materials

If the toy or any of its removable components fits entirely within the small parts cylinder (see EN 71-1) and contains accessible glass, ceramic or metallic materials then the toy or component shall be tested in accordance with 7.4.3.5 after removal of all accessible *coatings* in accordance with 7.3.3.1.

NOTE Inaccessible glass, ceramic and metallic *toy materials*, like other inaccessible materials, are not tested according to 7.4.3.5. Neither are accessible glass, ceramic and metallic toy components which do not fit within the small parts cylinder. The exposure to certain elements from these larger components, which cannot be swallowed, is not considered significant.

7.3.3.6 Other materials, whether mass coloured or not

If possible obtain a test portion of not less than 100 mg of the *toy material* according to 7.3.3.2, 7.3.3.3, 7.3.3.4 or 7.3.3.5 whichever is appropriate.

If a test portion of 100 mg or more cannot be obtained, a test portion shall be obtained from each *toy material* present in the laboratory sample in a mass greater than 10 mg. If the weight of the test portion obtained is between 10 mg and 100 mg, that weight shall be reported (see Clause 10 h)). In this case the analytical results shall be calculated as though 100 mg of the test portion had been used. If the *toy material* to be tested is coated with a *coating* of paint, varnish, lacquer, printing ink or similar *coating* follow the procedure in 7.3.3.1.

7.4 Migration procedure

7.4.1 General

The migration solution shall be used for the determination of elements. For the determinations of Chromium species (see H.8) and organic tin the migration solutions shall be stabilized and/or otherwise treated immediately after their preparation (see relevant analytical methods in Annex F and Annex G).

For other elements, if the migration solutions are to be retained for more than 24 h prior to analysis, they shall be stabilized by addition of hydrochloric acid so that the concentration HCl of the stored solution is approximately 1 mol/l. The stabilization by hydrochloric acid shall be reported (see Clause 10 h).

7.4.2 Category I: Dry, brittle, powder like or pliable and Category II: Liquid or sticky

7.4.2.1 Samples containing grease, oil, wax or similar material

Weigh, to the nearest 0,05 g, Wfp 2 × 25 g of water at approximately 20 °C into the extraction container containing the filter paper parcel. Macerate carefully without loss so that the filter paper is thoroughly soaked. Then add the same mass of 0,14 mol/l hydrochloric acid solution (6.1.2) at approximately 20 °C and mix. For the purposes of these measured additions the density of the water and hydrochloric acid solution can be assumed to be 1,0 g/ml and the solutions may be added volumetrically using suitable dispensers accurate to the nearest 0,05 ml. Record the weights or volumes of water and hydrochloric acid used (V_{H2O} and V_{HCl}).

〚A1〛 Shake the mixture for at least 1 min and measure the pH of the mixture. If the pH exceeds 1,30 add a solution of 2 mol/l hydrochloric acid (6.1.4) drop-wise whilst mixing until the pH is 1,2 ± 0,1. Close the container and agitate the mixture at (37 ± 2) °C (see 6.2.4) for 1 h. Stop the agitation and leave the containers to stand for a further 1 h at (37 ± 2) °C. 〚A1〛

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Without delay separate the solid matter from the solution by filtration through a membrane filter to remove visible solid material. Use filters with a pore size of 0,45 µm or 0,22 µm as necessary. In addition, if necessary, centrifuge at high speed (6.2.3) to remove remaining visible solid materials. Separation shall be completed as soon as possible after completion of the standing time; centrifuging shall take no longer than 10 min and shall be reported under Clause 10 h).

There is the possibility of small pigment particles passing through a 0,45 µm or 0,22 µm membrane filter (indicated by e.g. a cloudy solution, a Tyndall beam or a coloured filtrate) (see H.9). This could lead – in certain cases – to an incorrect determination of the migration value as the result measured will include not just the migrated element but also element chemically bound to the suspended particles. Where this occurs either refilter the solution immediately using a membrane filter of pore size 0,02 µm. Or where the refiltration cannot be performed, repeat the migration and separation steps finally using a membrane filter of pore size 0,02 µm.

7.4.2.2 Samples not containing grease, oil, wax or similar material

〚A1〛 Using the appropriate sized container (see 6.2.5), mix the test portion so prepared with 50 times its mass of an aqueous solution of 0,07 mol/l HCl (see 6.1.1) at approximately 20 °C. If appropriate, pliable modelling materials such as clay or doughy materials shall be suspended completely (e.g. by stirring). For a test portion mass of between 10 mg and 100 mg, mix the test portion with 5,0 ml of this solution. Shake for at least 1 min. Check the acidity of the mixture.

If the test portion is likely to contain large quantities of alkaline materials, generally in the form of calcium carbonate, adjust the pH to 1,2 ± 0,1 with approximately 6 mol/l HCl (see 6.1.5) in order to avoid over-dilution. The amount of hydrochloric acid used in relation to the amount of solution shall be reported under Clause 10 h). If only small quantities of alkaline material are present and the pH of the mixture is greater than 1,3 add drop-wise, while shaking the mixture, an aqueous solution of approximately 2 mol/l HCl (see 6.1.4) until the pH is 1,2 ± 0,1. Protect the mixture from light. Agitate the mixture at (37 ± 2) °C (see 6.2.4) for 1 h continuously and then allow to stand for 1 h at (37 ± 2) °C. 〚/A1〛

Without delay separate the solid matter from the solution by filtration through a membrane filter to remove visible solid material. Use filters with a pore size of 0,45 µm or 0,22 µm as necessary. In addition, if necessary, centrifuge at high speed (6.2.3) to remove remaining visible solid materials. Separation shall be completed as soon as possible after completion of the standing time; centrifuging shall take no longer than 10 min and shall be reported under Clause 10 h).

There is the possibility of small pigment particles passing through a 0,45 µm or 0,22 µm membrane filter (indicated by e.g. a cloudy solution, a Tyndall beam or a coloured filtrate) (see H.9). This could lead – in certain cases – to an incorrect determination of the migration value as the result measured will include not just the migrated element but also element chemically bound to the suspended particles. Where this occurs either refilter the solution immediately using a membrane filter of pore size 0,02 µm. Or where the refiltration cannot be performed, repeat the migration and separation steps finally using a membrane filter of pore size 0,02 µm.

7.4.3 Category III: Scraped-off

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

〚A1〛 Using the appropriate sized container (see 6.2.5), mix the test portion thus prepared with 50 times its mass of an aqueous solution of 0,07 mol/l HCl solution (see 6.1.1) at approximately 20 °C. Where the test portion has a mass of between 10 mg and 100 mg, mix the test portion with 5,0 ml of this solution. Shake for at least 1 min. If necessary, check the acidity of the mixture. If the pH is greater than 1,3 add drop-wise, while shaking the mixture, an aqueous solution of approximately 2 mol/l HCl (see 6.1.4) until the pH is 1,2 ± 0,1. Protect the mixture from light. Agitate the mixture at (37 ± 2) °C (see 6.2.4) for 1 h continuously and then allow to stand for 1 h at (37 ± 2) °C. 〚/A1〛

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Separate the solid matter from the solution by filtration through a membrane filter to remove visible solid material. Use filters with a pore size of 0,45 µm or 0,22 µm as necessary. In addition, if necessary, centrifuge at high speed (6.2.3) to remove remaining visible solid materials. Separation shall be completed as soon as possible after completion of the standing time; centrifuging shall take no longer than 10 min and shall be reported under Clause 10 h).

There is the possibility of small pigment particles passing through a 0,45 µm or 0,22 µm membrane filter (indicated by e.g. a cloudy solution, a Tyndall beam or a coloured filtrate) (see H.9). This could lead – in certain cases – to an incorrect determination of the migration value as the result measured will include not just the migrated element but also element chemically bound to the suspended particles. Where this occurs either refilter the solution immediately using a membrane filter of pore size 0,02 µm. Or where the refiltration cannot be performed, repeat the migration and separation steps finally using a membrane filter of pore size 0,02 µm.

7.4.3.2 Polymeric and similar materials including laminates and reinforced textile, but excluding other textiles

Follow the procedure in 7.4.3.1.

7.4.3.3 *Paper* and *paper board*

Macerate the test portion so prepared with 25 times its mass of water (see 6.1.7) so that the resulting mixture is homogeneous. Quantitatively transfer the mixture to the appropriate sized container (see 6.2.5). Add to the mixture an aqueous solution of 0,14 mol/l HCl (see 6.1.2) at approximately 20 °C at 25 times the mass of the test portion.

〚A1〛 Shake for at least 1 min. Check the acidity of the mixture. If the pH is greater than 1,3 add drop-wise, while shaking the mixture, an aqueous solution of approximately 2 mol/l HCl (see 6.1.4) until the pH is 1,2 ± 0,1. Protect the mixture from light. Agitate the mixture at (37 ± 2) °C (see 6.2.4) for 1 h continuously and then allow to stand for 1 h at (37 ± 2) °C. 〚A1〛

Without delay separate the solid matter from the solution by filtration through a membrane filter to remove visible solid material. Use filters with a pore size of 0,45 µm or 0,22 µm as necessary. In addition, if necessary, centrifuge at high speed (6.2.3) to remove remaining visible solid materials. Separation shall be completed as soon as possible after completion of the standing time; centrifuging shall take no longer than 10 min and shall be reported under Clause 10 h).

There is the possibility of small pigment particles passing through a 0,45 µm or 0,22 µm membrane filter (indicated by e.g. a cloudy solution, a Tyndall beam or a coloured filtrate) (see H.9). This could lead – in certain cases – to an incorrect determination of the migration value as the result measured will include not just the migrated element but also element chemically bound to the suspended particles. Where this occurs either refilter the solution immediately using a membrane filter of pore size 0,02 µm. Or where the refiltration cannot be performed, repeat the migration and separation steps finally using a membrane filter of pore size 0,02 µm.

7.4.3.4 Textiles, whether natural or synthetic

Follow the procedure in 7.4.3.1.

7.4.3.5 Glass, ceramic and metallic materials

〚A1〛 Weigh the toy or component into a 50 ml glass container with nominal dimensions: height 60 mm, diameter 40 mm. Add sufficient volume of an aqueous solution of 0,07 mol/l HCl (see 6.1.1) at approximately 20 °C to just cover the toy or component. Check the acidity of the mixture. If the pH is greater than 1,3 add drop-wise, while shaking the mixture, an aqueous solution of approximately 2 mol/l HCl (see 6.1.4) until the pH is 1,2 ± 0,1. Cover the container, protect the contents from light and allow the contents to stand for 2 h at (37 ± 2) °C. 〚A1〛

NOTE This type of container will take all components/toys that fit inside the small parts cylinder.

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Without delay separate the solid matter from the solution by filtration through a membrane filter to remove visible solid material. Use filters with a pore size of 0,45 µm or 0,22 µm as necessary. In addition, if necessary, centrifuge at high speed (6.2.3) to remove remaining visible solid materials. Separation shall be completed as soon as possible after completion of the standing time; centrifuging shall take no longer than 10 min and shall be reported under Clause 10 h).

There is the possibility of small pigment particles passing through a 0,45 µm or 0,22 µm membrane filter (indicated by e.g. a cloudy solution, a Tyndall beam or a coloured filtrate) (see H.9). This could lead – in certain cases – to an incorrect determination of the migration value as the result measured will include not just the migrated element but also element chemically bound to the suspended particles. Where this occurs either refilter the solution immediately using a membrane filter of pore size 0,02 µm. Or where the refiltration cannot be performed, repeat the migration and separation steps finally using a membrane filter of pore size 0,02 µm.

7.4.3.6 Other materials, whether mass coloured or not

The materials shall be tested by the most appropriate method of 7.4.3.2, 7.4.3.3, 7.4.3.4 or 7.4.3.5. The method used shall be reported under Clause 10 h).

8 Methods of analysis

For all types of sample preparation, the final extract is analysed for presence of elements by ICP-MS, ICP-OES, CVAAS, GC-MS or other suitable techniques. Examples of possible techniques are given in Annex E, Annex F and Annex G.

A validated method shall be used. The limits of quantification shall be below the limits given in Table 2.

The obtained migration solution (7.4) is analysed by three different methods (one for general elements, one for Chromium (III) and Chromium (VI) and one for organic tin). Each method requires an amount of the migration solution. For general elements 4 ml of migration solution is required, for Chromium (III) and (VI) 1 ml; for organic tin 5 ml. In order to determine all of the required elements, it may be necessary to perform an additional dilution of the migration solution. In this case, the limits of detection and the limits of quantification of the relevant analyses shall be reported under Clause 10 h).

9 Calculation of results

9.1 General elements

NOTE The term “general elements” refers to the elements Aluminium, Antimony, Arsenic, Barium, Boron, Cadmium, Chromium, Cobalt, Copper, Lead, Manganese, Mercury, Nickel, Selenium, Strontium, Tin and Zinc (see Clause 5).

9.1.1 Calibration curve

Prepare calibration curves of each element of interest by plotting the concentration of the standards of the element of interest (see 7.2) against the measuring signal. For quantification the calibration curve shall have a correlation coefficient better or equal to 0,990.

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9.1.2 Calculation of migration

Calculate the amount of an element in the sample by:

$$Migration(mg\ /\ kg) = \frac{C \cdot V \cdot 1\,000 \cdot f}{A}$$

where

Migration	: release of an element from the sample (mg/kg)
C	: concentration of the element in the migration solution (mg/l)
V	: volume of simulant added (ml)
A	: amount of test portion (mg)
f	: dilution factor

Report the results to the number of significant figures justified by the precision of the measurement.

9.1.3 Interpretation of results

The measurement uncertainty, $u_{t,r}$, shall be used to interpret an analytical result in relation to compliance assessment. Laboratories are required to determine their own performance data, including measurement uncertainty (see Annex B).

9.2 Chromium (III) and Chromium (VI)

9.2.1 Calibration curve

Prepare calibration curves of each element of interest by plotting the concentration of the standards of the element of interest (see 7.2.) against the measuring signal. For quantification, the calibration curve shall have a correlation coefficient better or equal to 0,990.

9.2.2 Calculation of migration

Calculate the amount of an element in the sample by:

$$Migration(mg\ /\ kg) = \frac{C \cdot V \cdot 1\,000 \cdot f}{A}$$

where

Migration	: release of an element from the sample (mg/kg)
C	: concentration of the element in the migration solution (mg/l)
V	: volume of simulant added (ml)
A	: amount of test portion (mg)
f	: dilution factor

NOTE For calculation of f the dilution factor needs to be taken into account caused by the neutralization step and the complexation step.

Report the results to the number of significant figures justified by the precision of the measurement.

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9.2.3 Interpretation of results

See 9.1.3.

9.3 Organic tin

9.3.1 Calibration curves

The procedure shall be calibrated using internal standards that are comparable to the organic tin compound of interest. In Annex G examples are given. For quantification the calibration curve shall have a correlation coefficient better or equal to 0,990.

9.3.2 Calculation of migration of single organic tin compound

For each detected organic tin cation the migration shall be calculated according to the following formula:

$$Migration(mg\ /\ kg) = \frac{C \cdot V \cdot R \cdot 1\,000 \cdot f}{A}$$

where

$Migration$	: release of an organic tin compound from the sample (mg/kg)
C	: concentration of the substance in the migration solution (mg/l)
V	: volume of simulant added (ml)
A	: amount of test portion (mg)
R	: relative molecular weight (see Table 3)
f	: dilution factor

NOTE When using the procedure described in Annex G, the concentration of the organic tin compound is already expressed as TBT as this is how the working solutions were prepared (see Table G.2). Therefore R in the formula does not need to be filled in. C in the formula corresponds to the concentration of the organic tin compound in the migration solution. The concentration of the organic tin compound in the hexane phase needs to be converted in to concentration in the migration solution.

A1

Table 3 — Molecular weight of organic tin cations

Organic tin cation	Molecular weight g/Mol	Relative molecular weight[a]
Methyl tin	133,7	2,169
Butyltin	175,8	1,650
Dibutyl tin	232,7	1,245
Tributyl tin	289,7	1,000
Tetrabutyl tin	346,7	0,835
n-Octyl tin	231,7	1,251
Di-n-octyl tin	344,7	0,840
Di-n-propyl tin	204,9	1,416
Diphenyl tin	272,7	1,063
Triphenyl tin	349,7	0,829
[a] Compared to tributyltin (see H.10).		

A1

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Organic tin compounds other than specified in Table 3 may also be present in toys. Table 3 contains a selection and is not a complete list of organic tin compounds.

9.3.3 Calculation of migration of organic tin

The migration of organic tin shall be calculated by adding the migration values for all the single organic tin compounds that have been detected and quantified. The migration of organic tin is expressed as tributyltin (see H.10).

9.3.4 Interpretation of results

See 9.1.3.

10 Test report

The test report shall at least contain:

a) a title, e.g. Test report, Test Certificate;

b) the name and address of the laboratory and the name and address of the client;

c) a serial number or similar unique identification for the report which should appear on each page together with pagination in the form Page .. of ..;

d) the client's identification details for the samples and the identifiers used by the laboratory, for example sample numbers;

e) the date of receipt of the items and the date(s) of testing;

f) identification of the analytical technique used;

g) a reference to this European Standard (i.e. A3 EN 71-3:2013+A3:2018 A3);

h) a note of any deviations from the standard and any environmental conditions which may bear upon the results;

i) the test results themselves with units and the measurement uncertainty if necessary;

j) the name, position and signature or other identification of the person accepting responsibility for the report and the report's date of issue;

k) a statement that the results only apply to the items tested.

NOTE For more information regarding the necessity of stating the measurement uncertainty, see Annex B and [6].

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Annex A
(informative)

Significant technical changes between this European Standard and the previous version

Table A.1

Clause/Paragraph/Table/Figure	Change
General	The standard has been revised to reflect new particular safety requirements in Directive 2009/48/EC, in comparison to 88/378/EEC
4. Requirements	The list of certain elements is expanded. Bioavailability has been replaced by migration limits in the new Directive. Migration limits are set for three different toy categories. The interpretation of results is moved to Clause 9. Table 2, containing analytical correction factors, is deleted. Instead, in Clause 9, measurement uncertainty is introduced (see also Annex B).
A1 6.2.2 equipment for measuring pH	The requirements on the equipment for measuring pH have been revised. A1
A1 7.4 Migration procedure	The migration conditions have been specified more precisely, in particular with regard to the pH value. A1
8. Preparation and analysis of test portions	It is now included in Clause 7. The text is redrafted. The principle is the same. The de-waxing procedure is updated.
A1 9.3 Organic tin	The (relative) molecular weights in Table 3 have been corrected. A1
10. Test report	The test report is now in line with EN ISO/IEC 17025.
Annex B	A new Annex B contains information on assessment of method performance and how to use measurement uncertainty.
Annex E	A new Annex E contains information on the method of analysis for general elements.
Annex F	A new Annex F contains information on the method of analysis for speciation of Chromium (III) and Chromium (VI).
A1 F.2.8 Hydrochloric acid solution	The instructions on how to prepare the hydrochloric acid solution have been deleted. A1
Annex G	A new Annex G contains information on the method of analysis for organic tin.
A1 G.3.22 Multi-component solutions and prepared reagents	Some of the values in Table G.2, e.g. some molecular weights, have been corrected. A1
A1 G.3.23 Hydrochloric acid solution	The instructions on how to prepare the hydrochloric acid solution have been deleted. A1

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A2 G.5.2	A note indicating that it is important to adapt the calibration solutions for the requirements of Category I and II accordingly has been added. A2
A2 G.6	Multiple Reaction Monitoring (MRM) mode (including the new Table G.4 and Figure G.1) has been added as alternative mode for the analysis. A2
A1 Annex H	The rationale has been revised. In particular, a cross-reference in H.7 has been corrected, the explanations in H.8 "Chromium (VI) migration", H.10 "Organic tin" and H.11 "pH value" have been revised and an additional subclause H.12 "De-waxing" has been added. A1
A2 H.10	The rationale on organic tin has been revised. In particular information on possible false-positive results has been added. A2
Annex I	A new Annex I contains information on conformity assessment other than by analysis.
Annex J	A new Annex J contains an informative list of organic tin compounds that may be present in toys but are not included in the validation of the method.
A1 Annex J	Dimethyltin bis(2-ethylhexylmercaptoacetate) has been deleted in Table J.1 and the families of some compounds have been corrected. A1
A1 Bibliography	An additional document [12] has been added to the Bibliography. A1
NOTE The technical changes referred include the significant technical changes from the EN revised but is not an exhaustive list of all modifications from the previous version.	

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Annex B
(informative)

Method performance

B.1 Method performance

A standardized method is defined as a by a group of users accepted and validated method. In case a laboratory introduces the standardized method without any changes, the laboratory shall demonstrate the results of their laboratory for important performance characteristics are in compliance with the validation results of the standardized method.

Validation includes specification of the requirements, determination of the characteristics of the method, a check that the requirements can be fulfilled by using the method, and a statement of validity.

This standardized method, migration process and analytical process, should be validated by using certified reference materials with known migration results for each element for a specific category. Since these certified reference materials are not available yet, the method is validated by using blank material and spiking the simulant after migration and filtration. To assess compliance the reproducibility and bias shall be determined. For reproducibility routine samples can be used and for determination of the bias representative certified reference material or reference material from a Round Robin with a consensus value can be used. Additionally the limit of detection and repeatability could be determined using routine samples. The CITAC / Eurachem Guide [5] can be used to determine these validation parameters.

B.2 Use of measurement uncertainty in compliance assessment

Clause 9 requires laboratories to determine their measurement uncertainty, $u_{t,r}$, and use this to interpret their analytical result in relation to compliance assessment. Guidance is given in the Eurachem/CITAC Guide [6].

The assessment is based on the concept of 'Decision rules'. These rules give a prescription for the acceptance or rejection of a product based on the measurement result, its uncertainty and the migration limit, taking into account the acceptable level of the probability of making a wrong decision. The application of a decision rule depends on the role of the laboratory, i.e. enforcement laboratory or (industrial) test laboratory.

A decision rule is that a result implies non compliance with the migration limit if the measured value exceeds the limit by the expanded uncertainty. This decision rule is usually applied by enforcement laboratories.

Another decision rule is that a result equal to or above the migration limit value implies non-compliance. A result below the limit, deduced by the uncertainty, implies compliance. This decision rule is usually applied by (industrial) test laboratories.

In this guide [6] several useful examples are given.

B.3 Information on round robin test

During the development of the revision of this European Standard a round robin test had been performed in spring 2012. Twenty-four laboratories applied the test method of the draft European Standard prEN 71-3:2012 on references materials provided by the lead laboratory responsible for the development of the test method. The reference materials covered the three material categories and all elements specified in Directive 2009/48/EC.

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The results on the different method part and on the 3 toy categories showed wide spread. Depending on the element/toy material category the statistical evaluation of this data lead to satisfying and unsatisfying estimations of the measurement uncertainty depending of the use of different statistical methods. These findings had been assessed by the CEN/TC 52/WG 5 “Safety of toys – Chemical properties” as not sufficient for taking the repeatability and reproducibility data on board of this standard. The evaluation of the causes for this is still ongoing.

Furthermore the following potentials for improvement had been identified:

— training of (some of) the laboratories;

— optimisations of reference materials, especially with reference to the used pigments, number of elements per material, long term stability for the 3 toy categories and the assessment of the limits e.g. for the Directive 2009/48/EC;

— accurate description of the procedures of sample preparation and migration;

— the procedures including equipment and instrumentation for the determination of the elements.

During the final conclusion for this version of the standard some modifications were introduced to improve the method where there were obvious reasons. For more improvement there is the intention to take the results during this revision into consideration, adapting the method and repeating the round robin test.

For the practical work of laboratories, there is the need of (certified) reference materials. Information on the availability of such reference materials will be given at http://www.toystandards.ds.dk under the section “Documents”.

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Annex C
(normative)

Sieve requirements

Table C.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 to exceed the nominal plus the figure)
0,500	0,315	+ 0,090	±0,018	+ 0,054

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Annex D
(informative)

Preparation and analysis of test portion

The diagram given below is an indication of which procedure to be used for the various *toy materials*.

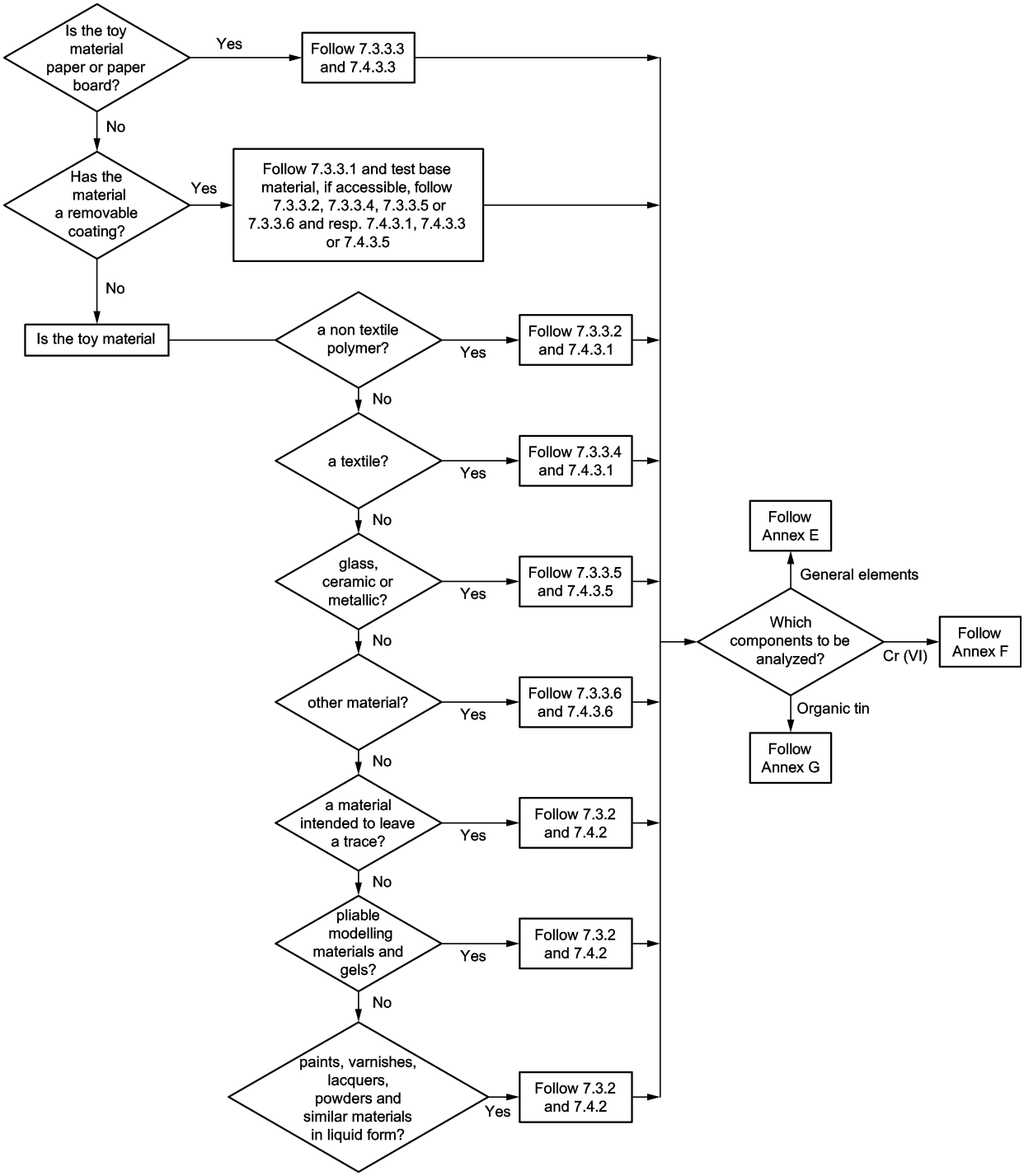


Figure D.1

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Annex E
(informative)

Method of analysis for general elements

E.1 Principle

This method quantifies Aluminium, Antimony, Arsenic, Barium, Boron, Cadmium, Chromium, Cobalt, Copper, Lead, Manganese, Mercury, Nickel, Selenium, Strontium, Tin and Zinc in the migration solution (7.4). The migration solution and its dilutions are analysed directly by ICP for identification and quantification.

E.2 Working solutions

E.2.1 Working solutions ICP-MS

E.2.1.1 Stock solution (M_1)

Prepare a stock solution (M_1) diluting the commercial available stock solutions of the individual elements (concentration = 1 000 mg/l) in hydrochloric acid solution (see 6.1.1) to a volume of 50 ml, according to the scheme in Table E.1.

Table E.1 — Preparation scheme stock solution M_1

Element	Volume stock ml	Volume ml	Concentration mg/l
Aluminium	0,5	50,0	10,0
Antimony	0,5	50,0	10,0
Arsenic	0,5	50,0	10,0
Barium	0,5	50,0	10,0
Boron	0,5	50,0	10,0
Cadmium	0,5	50,0	10,0
Chromium (III)[a]	0,5	50,0	10,0
Cobalt	0,5	50,0	10,0
Copper	0,5	50,0	10,0
Lead	0,5	50,0	10,0
Manganese	0,5	50,0	10,0
Mercury	0,05	50,0	1,0
Nickel	0,5	50,0	10,0
Selenium	0,5	50,0	10,0
Strontium	0,5	50,0	10,0
Tin	0,5	50,0	10,0
Zinc	0,5	50,0	10,0
[a] Chromium (III) is used to determine total Chromium, not for chromium (III) speciation.			

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E.2.1.2 Diluted stock solution (M_2)

Dilute 0,5 ml of stock solution M_1 in hydrochloric acid (see 6.1.1) to 50 ml.

E.2.1.3 Working solutions

Sequentially dilute diluted stock solutions (M_1) and (M_2) with hydrochloric acid solution (see 6.1.1), in order to obtain standards to be used for calibration according to the scheme in Table E.2.

Table E.2 — Preparation scheme working solutions

Working solutions	Volume M_1 ml	Volume M_2 ml	Volume HCl (6.1.1) ml	Volume total ml	Concentration of each element µg/l[a]
Working solution W_1		1,25	48,75	50,0	2,5
Working solution W_2		2,5	47,50	50,0	5,0
Working solution W_3		5,0	45,0	50,0	10,0
Working solution W_4	0,125		49,875	50,0	25,0
Working solution W_5	0,250		49,75	50,0	50,0
Working solution W_6	0,50		49,50	50,0	100,0
[a] The concentration of mercury is respectively 0,25, 0,50, 1,00, 2,5, 5,0 and 10,0 µg/l.					

E.2.1.4 Internal Standard stock solution

Prepare an Internal Standard stock solution diluting 250 µl of a commercial available stock solution containing Bismuth, Indium, Lithium and Yttrium (100 µg/ml in 7 % HNO_3 (volume fraction)) to a volume of 500 ml. This solution can either be used to prepare the standard and sample solutions, or by mixing with these solutions using continuous flow before entering the spray chamber.

E.2.2 Working solutions ICP-OES

E.2.2.1 Stock solution (M_1)

Commercial available stock solutions of the individual elements (see Table E.1) at the concentration of 1 000 mg/l.

E.2.2.2 Working solutions (calibration standards)

The working solutions are multi-elements solutions. Sequentially dilute stock solution of the individual elements with hydrochloric acid solution (see 6.1.1) to a final volume of 250 ml, in order to obtain standards to be used for calibration according to the scheme in Table E.3.

NOTE This working range is set for category III *toy materials.*

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Table E.3 — Preparation scheme working solutions

Working solutions	**Volume M_1** ml	**Volume total** ml	**Concentration of each elements** µg/l[a]
Working solution W_1	0,125	250,0	5
Working solution W_2	0,25	250,0	10
Working solution W_3	1,25	250,0	50
Working solution W_4	2,5	250,0	100
Working solution W_5	5,0	250,0	200
[a] The concentration of Mercury is respectively 0,50, 1,0, 5,0, 10,0, and 20,0 µg/l.			

E.3 Procedure

Use the migration solution (7.4). The elements shall be within the range of the calibration curve and therefore dilutions of the migration solutions may be necessary. For example a dilution range of 2, 20, 200 and 2 000 can be used.

E.4 Analysis

E.4.1 General

The migration solution can be analysed directly by ICP (Inductively Coupled Plasma) to determine the content of general elements (Aluminium, Antimony, Arsenic, Barium, Boron, Cadmium, Cobalt, Chromium, Copper, Lead, Manganese, Mercury, Nickel, Selenium, Strontium, Tin and Zinc). In Table E.4 ICP parameters are given that can be used.

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Table E.4 — ICP parameters

Parameter	Setting/Type	
	ICP-MS	ICP-OES
Nebuliser	High sensitivity quartz	Quartz Torch, high solids
Spray Chamber	Quartz Cyclonic	Quartz 'Sea Spray'
RF Power	1 600 W	1200–1 600 W, elem. Settings
Plasma Argon Flow	17 l/min	16,5 l/min
Nebuliser Argon Flow	1,02 l/min	0,75 l/min
Aux. Argon Flow	1,2 l/min	1,5 l/min
Injector	2,0 mm i.d. Quartz	2,3 mm i.d. Quartz
Mode	KED[a]	Simultaneous
Dwell Time	50 ms (per AMU)	20–30 s stabilizing
Total Acquisition Time	180 s	10–2-s measurement
CeO^+/Ce^+	< 2 %	
Gas	Helium	Argon
Gas Flow	4,4 ml/min	
RPq	0,45	

[a] For Boron and Aluminium it is preferred to use the Standard Mode.

NOTE For calculation and test report, 9.1 and Clause 10 need to be used.

E.4.2 Limit of detection and quantification

In Table E.5 typical limits of detection (LOD) and quantification (LOQ) for general elements are given when using ICP-MS.

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Table E.5 — Typical detection limits (LOD) and quantification limits (LOQ) expressed in mg/kg toy material

Element	**LOD** mg/kg	**LOQ** mg/kg
Aluminium	0,073	0,146
Antimony	0,014	0,029
Arsenic	0,027	0,055
Barium	0,027	0,054
Boron	0,039	0,078
Cadmium	0,059	0,118
Chromium (total)	0,023	0,046
Cobalt	0,019	0,039
Copper	0,010	0,020
Lead	0,061	0,122
Manganese	0,050	0,099
Mercury	0,010	0,021
Nickel	0,042	0,083
Selenium	0,147	0,294
Strontium	0,067	0,134
Tin	0,110	0,221
Zinc	0,097	0,197

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Annex F
(informative)

Methods of analysis for speciation of Chromium (III) and (VI)

F.1 Principle

This method quantifies Chromium (III) and Chromium (VI) in the migration solution (7.4). This method is capable to determine compliance with the migration limit for category III *toy materials*. The limit of quantification is higher than the migration limits for category I and II *toy materials*. In I.2 guidance is given how compliance can be demonstrated for category I and II *toy materials*.

To prevent interconversion of Chromium (III) and Chromium (VI), the migration solution is neutralized directly after the migration step. Experiments have shown that at a pH of 7,1 both species are stable. Mobile phase is added, containing EDTA. EDTA reacts to form a complex with Chromium (III). Chromium (III) and Chromium (VI) are then separated by liquid chromatography using an LC-ICP-MS technique.

When analysing Chromium (III) and (VI) in one run, a high peak of Chromium (III) may influence the peak of Chromium (VI). This can be solved by skipping the complexation step with EDTA, thereby only showing the Chromium (VI) peak in the chromatogram. Chromium (III) can then be determined by either analysing the migration solution as well after the complexation step, or by determining the migration of total Chromium: Chromium (III) = total Chromium – Chromium (VI).

F.2 Reagents

Use reagents of highest purity.

F.2.1 Water, Milli Q 18,2 MΩ.cm.

F.2.2 Hydrochloric acid, HCl (30 % d = 1,15 g/ml or equal).

F.2.3 Tetrabutylammonium hydroxide (TBAH), 40 wt % in water, $[CH_3(CH_2)_3]_4NOH$, CAS# 2052-49-5.

F.2.4 Potassium Ethylenediamine tetraacetic acid, $C_{10}H_{14}K_2N_2O_8 * 2H_2O$, CAS# 7379-27-3.

F.2.5 Ammonia, NH_3 (25 % in water).

F.2.5.1 Ammonia solution, c(NH_3 (aq) = (0,07 ± 0,005) mol/l.

Add 5,3 ml ammonia solution (F.2.5) into a 1 000 ml volumetric flask. Make up to the mark with water (F.2.1) and mix carefully.

F.2.6 Acetic acid (glacial), 100 %.

F.2.7 Methanol, CH_3OH.

〖A1〉

F.2.8 Hydrochloric acid solution, c(HCl) = (0,07 ± 0,005) mol/l. 〈A1〗

F.2.9 Chromium (III) 1 000 mg/l in water (F.2.1).

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F.2.10 Chromium (VI) 1 000 mg/l in water (F.2.1).

F.2.11 Stock solution Chromium (M_1).

Prepare a stock solution (M_1) diluting 0,5 ml of the commercial available stock solutions of the individual elements (concentration = 1 000 mg/l) in mobile phase (F.2.14) to a volume of 50 ml. Allow the final solution to stand for 1 h at 50 °C.

F.2.12 Diluted stock solution (M_2).

Dilute 0,5 ml of stock solution M_1 in mobile phase (F.2.14) to 50 ml.

F.2.13 Working solutions (Calibrating Standards).

Sequentially dilute diluted stock solution (M_2) with mobile phase (F.2.14), after addition of 2,5 ml ammonia (F.2.5.1) and 2,5 ml hydrochloric acid (F.2.8), in order to obtain standards to be used for calibration according to the scheme in Table F.1. Allow the working solutions to stand for 1 h at 50 °C.

Table F.1 — Preparation scheme working solutions

Working solutions	Volume M_2 µl	Volume total ml	Concentration of each element µg/l
Working solution W_1	50	25,0	0,2
Working solution W_2	100	25,0	0,4
Working solution W_3	150	25,0	0,6
Working solution W_4	200	25,0	0,8
Working solution W_5	250	25,0	1,0
Working solution W_6	500	25,0	2,0

F.2.14 Mobile phase

Weigh, to the nearest 1 mg into a 1 000 ml volumetric flask approximately 647 mg TBAH (F.2.3) and 243 mg EDTA (F.2.4) and dissolve in water (F.2.1). Adjust the pH with acetic acid to 7,1.

F.3Apparatus

F.3.1 Volumetric flasks, glass.

F.3.2 Variable pipettes with disposable tips, variable range.

F.3.3 ICP-MS, equipped with HPLC module.

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F.3.4 pH-meter.

F.4Procedure

Use 1 ml of migration solution (7.4). Add 1 ml ammonia (F.2.5.1) to 1 ml migration solution. Additionally 8 ml mobile phase solution (F.2.14) is added. Allow the final solution to stand for 1 h at 50 °C.

〚A1〉 NOTE 1When the pH was adjusted (7.4.2), the amount of ammonia added needs to be equivalent to the total amount of HCl. The pH of 7,1 is established after addition of 8 ml of mobile phase. This needs to be adjusted as required. 〈A1〛

NOTE 2 It has been shown that presence of Manganese (VII) will lead to oxidation of Chromium (III) into Chromium (VI).

F.5Analysis

F.5.1 General

Inject samples and calibration solutions into the LC-ICP-MS system.

The following chromatographic and ICP-MS (F.5.1) settings can be used for the quantification of Chromium (III) and Chromium (VI).

F.5.2 LC-ICP-MS conditions

Table F.2 — Chromatographic settings for LC-ICP-MS

Parameter	Setting
Mobile phase	1 mM Tetrabutylammonium Hydroxide (TBAOH) + 0,6 mM EDTA (potassium salt); pH 7,1. Then added 2 % (volume fraction) MeOH in water
Flow rate	1,5 ml/min
Run time	3 min
Column	C8, monofunctional base deactivated, 3 µm, 33 × 4,6 mm
Column temperature	Ambient
Autosampler flush solvent	2 % methanol/〚A2〉 98 〈A2〛 % HPLC-grade water
Sample injection volume	50 µl
Sample prep	Dilute with mobile phase
Detection	ICP-MS
Total analysis time	3 min

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Table F.3 — ICP-MS parameters (Chromium (III) and (VI))

Parameter	ICP-MS
Nebuliser	High sensitivity quartz
Spray Chamber	Quartz Cyclonic
RF Power	1 600 W
Plasma Argon Flow	15 l/min
Nebuliser Argon Flow	0,96 l/min
Aux. Argon Flow	1,2 l/min
Injector	2,0 mm i.d. Quartz
Mode	DRC
Dwell Time	1 000 ms
Total Acquisition Time	180 s
CeO^+/Ce^+	< 2 %
Gas	NH_3
Gas Flow	0,50 ml/min
RPq	0,70

NOTE For calculation and test report, 9.2 and Clause 10 need to be used.

F.5.3 Limit of detection and quantification

F.5.3.1 General

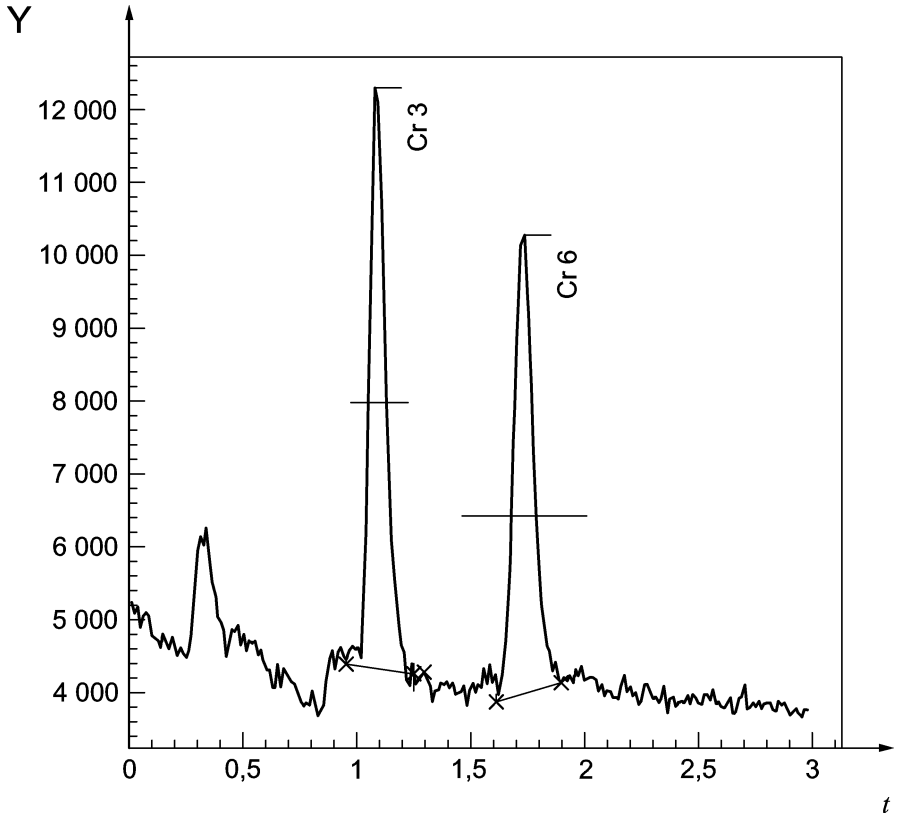
In Table F.4 typical limits of detection (LOD) and quantification (LOQ) are given.

Table F.4 — Typical detection limit (LOD) and quantification limit (LOQ) expressed in mg/kg toy material

Element	LOD mg/kg	LOQ mg/kg
Chromium (III)	0,064	0,128
Chromium (VI)	0,026	0,053

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F.5.3.2 Example of chromatogram



Key

Y intensity, in cps

t time, in min

Figure F.1 — Example of chromatogram of 0,6 ppb Chromium (III) and (VI)

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Annex G
(informative)

Method of analysis for organic tin (see H.10)

G.1 Principle

This method quantifies organic tin compounds and/or cations as mentioned in Table G.1 in the migration solution (7.4). The list of compounds in Table G.1 is a selection and not complete. Other organic tin compounds may also be present in toys (see Annex J). The anionic part bound to the organic tin cation is mainly dependent on the chemical environment and is not determined using this method.

Table G.1 — Organic tin compounds and cations

$R_nSn(4-n)^+$	R	*n*	Name	Acronym
$MeSn^{3+}$	Methyl	1	Methyltin cation	MeT
$BuSn^{3+}$	Butyl	1	Butyl cation	BuT
Bu_2Sn^{2+}	Butyl	2	Dibutyltin cation	DBT
Bu_3Sn^{+}	Butyl	3	Tributyltin cation	TBT
Bu_4Sn	Butyl	4	Tetrabutyltin cation	TeBT
$OcSn^{3+}$	Octyl	1	Monooctyltin cation	MOT
Oc_2Sn^{2+}	Octyl	2	Dioctyltin cation	DOT
Pro_2Sn^{2+}	Propyl	2	Dipropyltin cation	DProT
Ph_2Sn^{2+}	Phenyl	2	Diphenyltin cation	DPhT
Ph_3Sn^{+}	Phenyl	3	Triphenyltin cation	TPhT

Organic tin compounds are substances which contain an organic moiety in their structure. Organic tin compounds can only be determined in accordance with the standard after derivatisation.

Most organic tin compounds are too polar to be analysed directly by gas-chromatography and shall be derivatised to form non-polar alkyltin compounds prior to analysis.

The per-alkylated organic tin compounds behave in a completely different way from their parent compounds. Tetra-alkylated organic tin compounds which are already per-alkylated, such as tetrabutyltin, are determined directly without derivatisation.

This standard method utilizes *in situ* derivatisation using tetraethylborate to produce ethyl organic tin derivatives. The ethylated derivatives are extracted with hexane and analysed by GC-MS in full scan mode. For quantification single ion mode is preferred.

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The concentration is determined by calibration for the total procedure using an internal standard mixture. The internal standards shall be comparable to the organic tin compound of interest. Tributyl tin (d27) is the internal standard for the determination of Methyl tin, Butyltin, Di-n-propyl tin, Dibutyl tin, Tributyl tin, n-Octyl tin and Di-n-octyl tin. Tetrabutyl tin (d36) is the internal standard for the determination of Tetrabutyl tin. Triphenyl tin (d15) is the internal standard for the determination of Diphenyl tin and Triphenyl tin.

G.2 Terms and definitions

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

G.2.1
Organic Tin Compound
OTC
substance with at least one Sn-C bond

Note 1 to entry: The number of Sn-C bonds is a measure of the degree of substitution.

G.2.2
Organic Tin Cation
OC
part of the organic tin compound that contains all Sn-C bonds (and which is formally loaded)

Note 1 to entry: In this standard, the abbreviation OC is also used for the non-dissociated tetra-substituted organic tin. OC therefore comprises the cations MeT, DBT, TBT, TeBT, MOT, DOT, DProT, DPhT and TPhT.

G.3 Reagents

Use reagents of highest purity.

G.3.1 Water, free of substances causing interference with this method.

G.3.2 Hydrochloric acid, HCl (37 %).

G.3.3 Acetic acid, CH_3COOH, glacial.

G.3.4 Sodium acetate, 3 aq, $CH_3COONa.3H2O$.

G.3.5 Sodium sulfate, Na_2SO_4, anhydrous.

G.3.6 Methanol, CH_3OH.

G.3.7 Hexane, C_6H_{14}.

G.3.8 Sodium tetraethylborate, $NaB(C_2H_5)_4$, CAS# 15523-24-7.

G.3.9 Methyltin trichloride, MeTCl, CH_3SnCl_3, CAS# 993-16-8.

G.3.10 Butyltin trichloride, BuTCl, C4H9SnCl3, CAS# 1118-46-3.

G.3.11 Dibutyltin dichloride, DBTCl, $(C_4H_9)_2SnCl_2$, CAS# 683-18-1.

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G.3.12 Tributyltin chloride, TBTCl, $(C_4H_9)_3SnCl$, CAS# 1461-22-9.

G.3.13 Tetrabutyltin, TeBT, $(C_4H_9)_4Sn$,CAS# 1461-25-2.

G.3.14 Monooctyltin trichloride, MOTCl, $C_8H_{17}SnCl_3$, CAS# 3091-25-6.

G.3.15 Dioctyltin dichloride, DOTCl, $(C_8H_{17})_2SnCl_2$, CAS# 3542-36-7.

G.3.16 Dipropyltin dichloride, DProTCl, $(C_3H_6)_2SnCl_2$, CAS# 867-36-7.

G.3.17 Diphenyltin dichloride, DPhTCl, $(C_6H_5)_2SnCl_2$, CAS# 1135-99-5.

G.3.18 Triphenyltin chloride, TPhTCl, $(C_6H_5)_3SnCl$, CAS# 639-58-7.

G.3.19 Tributyl-d27-tin chloride, TBTTCL (d27), $(C_4D_9)_3SnCl$, CAS# 1257647-76-9.

G.3.20 Tetrabutyl-d36-tin, TeBT (d36), $(C_4D_9)_4Sn$, CAS# 358731-92-7.

G.3.21 Triphenyl-d15-tin chloride, TPhTCl (d15), $(C_6D_5)_3SnCl$, CAS# 358731-94-9.

G.3.22 Multi-component solutions and prepared reagents.

G.3.22.1 General.

Since stability of multi-component standard solutions is a matter of concern, it is recommended to prepare several solutions containing solely organic tin compounds with the same degree of alkylation/arylation (e.g. four solutions respectively for mono-, di-, tri-, and tetra-substituted compounds). Stability can be assessed by the absence of degradation products.

G.3.22.2 Multi-component-standard solution in methanol, Stock solution A.

For the preparation of 1 000 mg/l of organic tin cation stock solution, weigh, to the nearest 0,1 mg, the amounts of organic tin compounds specified in Table G.2 into a 100 ml volumetric flask. Dissolve these compounds in a small amount of methanol (G.3.6). Then make up to the mark with methanol and mix carefully.

If stored at 4 °C in the dark, the solution is stable for up to one year.

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Table G.2 — Required portions of organic tin compounds, and their weighing factors, corresponding to 100 mg of organic tin cations (expressed as TBT cation)

Substance ($OTCl_x$)	Weighed portion mg	MW $OTCl_x$	MW OC	Weighing factor [a]	Organic tin cation	Relative molecular weight [b]
Methyl tin trichloride	82,8	240,1	133,7	0,557	MeT	2,169
Di-n-propyl tin dichloride	95,1	275,8	204,9	0,743	DProT	1,416
Butyl tin trichloride	97,4	282,2	175,8	0,623	BuT	1,650
Dibutyl tin dichloride	104,7	303,6	232,7	0,767	DBT	1,245
Tributyl tin chloride	112,2	325,2	289,7	0,891	TBT	1,000
n-Octyl tin trichloride	116,6	338,1	231,7	0,686	MOT	1,251
Di-n-octyl tin dichloride	143,4	415,6	344,7	0,830	DOT	0,840
Tetrabutyl tin	119,6	346,7	346,7	1,000	TeBT	0,835
Diphenyl tin dichloride	118,6	343,6	272,7	0,794	DPhT	1,063
Triphenyl tin chloride	132,9	385,2	349,7	0,908	TPhT	0,829

[a] Weighing factor = molar mass (organic tin cation)/molar mass (organic tin chloride).

[b] Compared to tributyltin.

A1

G.3.22.3 Calibration solution of OC, working solution B.

Dilute from the stock solution A (G.3.22.2) to a concentration of 10 mg of each OC (expressed as TBT cation) per litre in methanol (dilution factor 100).

G.3.22.4 Mix solution of the internal standards in methanol, internal standard stock solution C.

Weigh, to the nearest 0,1 mg, into a 100 ml volumetric flask, approximately 100 mg of Tributyl-d27-tin chloride (G.3.19), 100 mg of Tetrabutyl-d36-tin (G.3.20) and 100 mg of Triphenyl-d15-tin chloride (G.3.21) and dissolve in a small amount of methanol (G.3.6). Make up to the mark with methanol and mix carefully.

The solution is stable for up to one year when stored in the dark at 4 °C.

G.3.22.5 Working solution of the internal standards in methanol, internal standard working solution D (2 mg deuterated OCT per litre in methanol).

Pipette 0,2 ml of the internal standard stock solution C into a 100 ml volumetric flask. Make up to the mark with methanol and mix carefully.

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[A1]

G.3.23 Hydrochloric acid solution, c(HCl) = (0,07 ± 0,005) mol/l. [A1]

G.3.24 Acetate buffer solution.

Dissolve about 16,6 g sodium acetate.3aq (G.3.4) in 250 ml of water (G.3.1) into a 500 ml volumetric flask. Add 1,2 ml glacial acetic acid (G.3.3) to reach a pH of [A2] 4,5 [A2]. Make up to the mark with water (G.3.1) and mix carefully.

G.3.25 Derivatisation agent (2 % mass concentration in water).

Weigh about 200 mg of sodium tetraethylborate (G.3.8) into a 10 ml volumetric flask and make up to volume with water (G.3.1). This solution is not stable, and shall be prepared daily fresh.

NOTE Caution derivatisation agent is flammable.

G.4 Apparatus

NOTE 1 Care needs to be exercised to avoid contamination. Therefore flasks, equipment and anything making contact with test samples and extracts thereof, need to be dedicated. Cleaning of dishwashers needs to be avoided. Instead rinsing with ethanol and hexane is preferred.

NOTE 2 To minimize recovery losses by adsorption, the use of polypropylene recipients instead of glass is preferred.

NOTE 3 It is important to verify blank values and recovery.

G.4.1 Volumetric flasks, of 10 ml, 50 ml, 100 ml and 1 000 ml.

G.4.2 Variable pipettes for organic fluids with disposable tips, with a volume range from 10 to 100 µl, 100 to 1000 µl and 1 to 5 ml.

G.4.3 Disposable 50 ml, propylene conical tube with screw cap.

G.4.4 Multi tube vortex.

G.4.5 Gas chromatograph, equipped with split/splitless injector.

G.4.6 Autosampler device.

G.4.7 Mass spectrometer for electron impact (EI) mode.

G.5 Procedure

G.5.1 Sample derivatisation

Use 5 ml of migration solution (7.4). Add 0,1 ml internal standard solution D (G.3.22.5), 5 ml of acetate buffer (G.3.24) is added to the migration solution to adjust the pH to 4,7. Then 0,5 ml of 2 % sodium tetra ethyl borate (G.3.25) and 2 ml of hexane (G.3.7) are added. The mixture is shaken for 30 min with a vortex (G.4.4) and left to stand until the phase separation is complete. The hexane fraction is then analysed by GC-MS.

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G.5.2 Calibration standards

Take 7 disposable 50 ml tubes, add to each tube 5 ml gastric simulant (G.3.23) and add to each tube with a pipette (G.4.2) respectively 0, 20 µl, 50 µl, 100 µl, 0,20 ml, 0,50 ml and 1,5 ml working solution B (G.3.22.3). Add 0,1 ml internal standard solution D (G.3.22.5), 5 ml of acetate buffer (G.3.24) is added to the resulting migration solution to adjust the pH to 4,7. Then 0,5 ml of 2 % sodium tetra ethyl borate (G.3.25) and 2 ml of hexane (G.3.7) are added. The mixture is shaken for 30 min with a vortex (G.4.4) and left to stand until the phase separation is complete. The hexane fraction is then analysed by GC-MS.

[A2] NOTE It is important that calibration solutions for the requirements of Category I and II are adapted accordingly. [A2]

G.6 Analysis

G.6.1 General

Optimize the instrument (G.4.5) according to the manufacturer's instructions.

Analyse the analytes in the Selective Ion Monitoring (SIM) mode (Table G.3) [A2] or Multiple Reaction Monitoring (MRM) mode (see Table G.4 and Figure G.1 for examples). [A2]

Table G.3 — Selected ion monitoring for organic tin cations

Organic tin Ethyl derivative	**Abbreviation**	**m/z**
Methyl tin	MeT	163,165,191,193
Butyl tin	BuT	121, 149, 179, 235
Di-n-propyl tin	DProT	191,193,233,235
Dibutyl tin	DBT	205,207,261,263
Tributyl tin	TBT	205,207,289,291
n-Octyl tin	MOT	177,179,289,291
Tetrabutyl tin	TeBT	177,179,289,291
Diphenyl tin	DPhT	195,301,305
Di-n-octyl tin	DOT	261,263,307,375
Triphenyl tin	TPhT	347,349,351
Internal Standards		
Tributyl tin (d27)	TBT (d27)	217,281,318
Tetrabutyl tin (d36)	TeBT (d36)	190,254,318
Triphenyl tin (d15)	TPhT (d15)	364,366

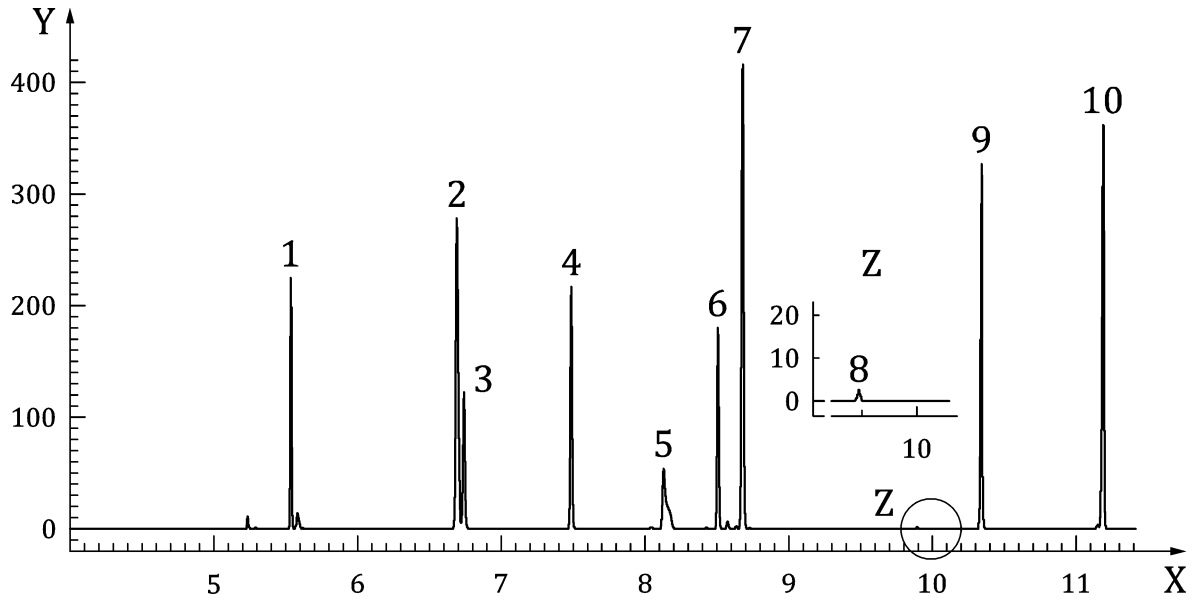
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A2

Table G.4 — Example of MRM data

Analyte	Precursor ion	Quantification ion	Collision energy of quantification ion	Qualification ion 1	Collision energy of qualification ion 1	Qualification ion 2	Collision energy of qualification ion 2
MeT	193	165	5	137	10		
DProT	235	151	5	193	5	123	20
MBT (BuT)	235	151	10	179	5		
DBT	263	151	10	123	25		
TBT-d27	318	190	20	254	7,5		
TBT	291	179	10	235	5		
MOT	291	179	8				
	179			151	10		
TeBT-d36	318	190	10	254	7,5		
TeBT	291	179	10				
	235			123	7		
DPhT	303	197	20	275	10		
DOT	263	151	10	123	10		
TPhT-d15	366	120	25	201	20		
TPhT	351	197	20	120	20		

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Key

X:	Time, min	5:	TBT/TBT-d27
Y:	Intensity, MCps	6:	MOT
1:	MeT	7:	TeBT/TeBT-d36
2:	DProT	8:	DPhT
3:	MBT (BuT)	9:	DOT
4:	DBT	10:	TPhT/TPhT-d15

Figure G.1 — Example of MRM Chromatogram

A2

Quantify the gas chromatographic signals. The evaluation using peak areas is recommended.

The injection sequence is as follows:

— hexane;

— blank extract;

— calibration solutions;

— hexane;

— sample extracts.

Examples of GC and MS conditions are given in G.6.1 and G.6.2 respectively.

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G.6.2 Example of GC conditions

Injection conditions:	Pulsed splitless, pulse pressure = 30 kPa, pulse time = 0,5 min
	Purge flow = 50 ml/min, purge time = 2,00 min
Injection temperature:	275 °C
Carrier:	Helium; Constant flow: 1,7 ml/min
Column:	5 %-diphenyl-95 %-dimethyl polysiloxane copolymer, 30 m x 0,32 mm ID x
	0,25 µm film thickness
Oven temperature program:	50 °C (1 min) - 20 °C/min - 280 °C (2,5 min)
Injection volume:	2 µl

G.6.3 Example of MS conditions

Transfer line:	280 °C
Electron impact:	70 eV
Electron multiplier:	source dependent
Selected ion monitoring mode:	choose at least three of the most intensive non-interfered fragment ions

NOTE For calculation, Subclause 9.3 needs to be used. The concentration of the organic tin compound is already expressed as TBT as this how the working solutions were prepared (see Table G.2). Therefore R in the formula does not need to be filled in. C in the formula corresponds to the concentration of the organic tin compound in the migration solution. The concentration of the organic tin compound in the hexane phase needs to be converted in to concentration in the migration solution. For the test report, Clause 10 needs to be used.

G.6.4 Limit of detection and quantification

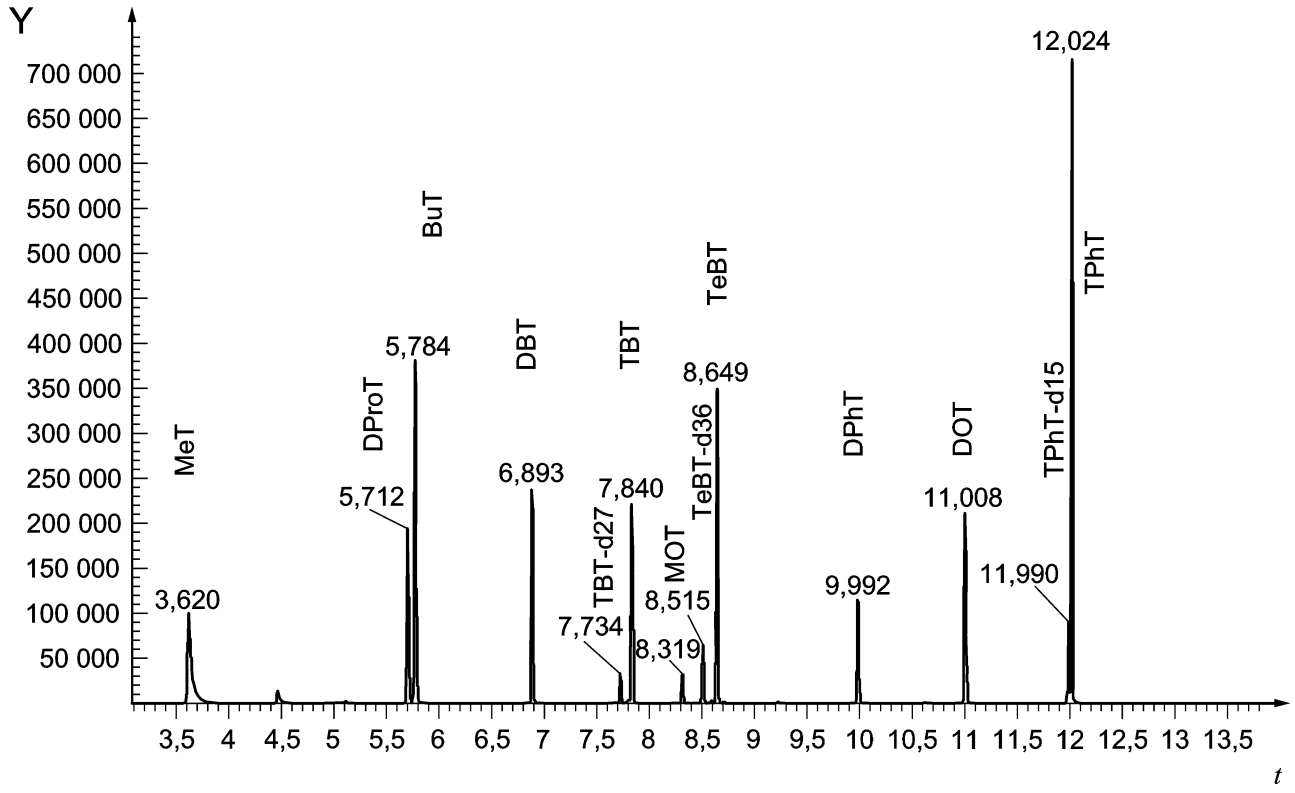
In Table A2 G.5 A2 typical limits of detection (LOD) and quantification (LOQ) for organic tin compounds are given.

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Table [A2] G.5 [A2] — Typical detection limits (LOD) and quantification limits (LOQ) expressed in mg/kg toy material

Element	LOD mg/kg	LOQ mg/kg
Methyltin	0,06	0,12
Butyltin	0,08	0,16
Tributyltin	0,12	0,24
Monooctyltin	0,37	0,74
Dioctyltin	0,17	0,35
Dibutyltin	0,08	0,16
Dipropyltin	0,26	0,52
Tetrabutyltin	0,12	0,24
Diphenyltin	0,54	1,08
Triphenyltin	0,15	0,29

G.6.5 Example of a GC-MS chromatogram



Key

Y abundance

t time, in min

Figure [A2] G.2 [A2] — Example of a GC-MS chromatogram of ethylated organic tin compounds

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Annex H
(informative)

Rationale

H.1 General

Migration limits for certain elements in toy materials or parts of toys are set in Annex II, III.13 of the Toy Safety Directive 2009/48/EC [1]. The limits in the Toy Safety Directive have been influenced by an RIVM report according to the criteria of the relevant Scientific Committee, in order to ensure that these elements should be present at safe levels for children and compatible with good manufacturing practice. This assessment includes a differentiation of toys into three toy categories, based on different assumed consumption. The migration limits do not apply to toys or components of toys which, due to their accessibility, function, volume or mass, clearly exclude any hazard due to sucking, licking, swallowing or prolonged contact with skin, bearing in mind the normal and foreseeable behaviour of children.

This standard specifies the sampling, sample preparation and migration procedure. The migration procedure simulates contact of the toy material with gastric juices for a period of time after swallowing.

The sample preparation and migration procedure are given in the normative part of this standard (Clause 7). In Clause 8 performance criteria are given for the analytical techniques to determine the amount of a certain element in the resulting migration solution. In the informative Annex E, Annex F and Annex G the standard preparation and analytical methods are given. These methods have been developed and validated by the lead laboratory, and were confirmed by the peer review laboratory. Other standards preparation and analytical techniques may also be used, as long as they meet the requirements specified in Clause 8.

In the informative Annex B requirements are given for laboratories how to establish their method performance, including measurement uncertainty. The measurement uncertainty shall be applied in order to interpret the result in relation to compliance assessment. In Annex I further guidance is given how to assess conformity with respect to certain elements.

H.2 Mouthing behaviour of children (Scope)

Most studies on mouthing behaviour look at children under three years old. Only 3 studies are known that observe children over three years of age [8,9,10], of which only one presents mouthing duration data [8]. The mean mouthing time on toys for 4 and 5 year old children was very low (3 and 1 min per day respectively) with a maximum observed mouthing time on toys of 20 and 11 min per day.

By the age of 6 years, children do not mouth their toys to any significant degree.

H.3 Skin contact (Scope)

The RIVM has conducted a study on the assessment methodology of chemical safety of toys with a focus on elements and identified the oral exposure to elements as the relevant route [7]. Most if not all toys or accessible parts of toys will come in contact with the skin, which may result in dermal exposure and via hand to mouth contact in additional oral exposure. For systemic exposure to elements, the dermal exposure route is less relevant than the oral exposure route, as the dermal absorption of elements is distinctly lower as their oral absorption. Therefore as a worst case simulant hydrochloric acid was chosen as gastric simulant for migration, also covering other exposure routes.

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H.4 Toy categories (4.1)

Directive 2009/48/EC [1] sets migration limits for certain elements in three different toy categories, based on the assumed ingestion of the materials. Further explanation of the categories is given in the Explanatory Guidance Document [3].

Category I: Dry, brittle, powder like or pliable

Category I includes solid toy material from which powder-like material is released during play. The material can be ingested. Contamination of the hands with powder contributes to enhanced oral exposure. The assumed ingestion is 100 mg per day.

Category II: Liquid or sticky

Category II includes fluid or viscous toy material which can be ingested and/or to which dermal exposure occurs during playing. The assumed ingestion is 400 mg per day.

Category III: Scraped-off

Category III includes solid toy material with or without a coating which can be ingested as a result of biting, tooth scraping, sucking or licking. This category includes those materials which are not covered by category I and II. The assumed ingestion is 8 mg per day.

H.5 Test portions (7.1, 7.3.3.2, 7.3.3.3, 7.3.3.6)

For category III toys (Scraped off) it is not always possible to obtain 100 mg of *toy material*.

If less than 10 mg of *toy material* can be obtained, it is not required to analyse. From such a small amount of *toy material* the exposure to certain elements is considered to be negligible.

If 10 mg to 100 mg of *toy material* is available, than the test shall be performed as if it were 100 mg of material. The migration is calculated as though 100 mg of the test portion had been used. This compensates for the low exposure to certain elements by migration from such low amounts of *toy material*.

H.6 Size of test pieces (7.3.3.2, 7.3.3.3, 7.3.3.4)

The test pieces should have at least one dimension of approximately 6 mm, but preferable two dimensions. As an example, a paper sheet shall be cut in pieces of approximately 6 by 6 mm. A fish line shall be cut in pieces of approximately 6 mm in length. It shall be practical for the laboratory to prepare these pieces. The aim is to achieve comparable surface areas between laboratories.

H.7 Textile threads

〚A1〛 Sewing threads, embroidery threads and other accessible threads are included in 7.3.3.4. Monofilament threads are not included in this subclause, but in 7.3.3.2. 〚A1〛

H.8 Chromium (VI) migration (7.4)

The migration of Chromium (VI) was tested in different migration simulants: Milli Q water; tap water; gastric simulant. In MiliQ water there is hardly any migration of Chromium (VI). A little migration occurs in tap water. The highest migration was observed in the gastric stimulant. It was therefore decided to keep the gastric simulant also for the migration procedure for Chromium (III) and (VI).

Interconversion of Chromium (III) and Chromium (VI) may occur. This equilibrium is influenced by the pH. Therefore immediately after the migration procedure the migration solution shall be stabilized by performing a neutralization step. Experiments have shown that at a pH of 7,1 both Chromium species are stable for at least 4 h (normal analysis time).

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The method in Annex F is not sufficiently sensitive for category I and II toy materials. This is due to the Chromium VI chemistry and the matrix effect. In I.2 guidance is given how to assess compliance for category I and II toy materials.

[A1] Alternative analytical techniques such as IC-UV-Vis, UV-Vis Spectrophotometer with integrating sphere or IC-ICP-MS, may also be used as long as they meet the requirements of Clause 8 and Clause 9. [A1]

H.9 Filtration of migration solutions (7.4.2.1, 7.4.2.2, 7.4.3.1, 7.4.3.3, 7.4.3.5)

Certain pigments may pass through filters with pore size 0,45 µm and 0,22 µm and thus lead to overestimation of the element migration analysis. Pigments in the filtrate will be indicated by a coloured filtrate.

If the filtrate is coloured after passing through the 0,22 µm filter either it should be immediately passed through a 0,02 µm membrane filter before the analytical measurement or the migration step should be repeated because of alterations in the filtrate.

H.10 Organic tin (9.3)

[A1] The requirement in the Toy Safety Directive is for total migratable organic tin. The method in Annex G determines the migration of individual organic tin compounds. The method was validated using a list of organic tin compounds that was selected for this purpose by the Working Group (CEN TC 52/ WG 5). Selection was based on the toxicity and exposure (presence in toys). Other organic tin compounds may also be present in toys.

EFSA has published an opinion on oral exposure of consumers to organotins [11]. The group TDI for organotins is expressed either as Sn content or as TBT chloride. For this European Standard it was chosen to express the migration of individual organotins as TBT, as the chloride is removed during the derivatisation step.

The test method has been validated for material category III. When testing materials of categories I and II (although it is not expected for organotin to be found in category I or II materials) the test methods need to be validated for these rare cases. For alternative options see I.1. [A1]

[A2] The laboratory should take care of the quality and purity of the derivatisation reagent.

Recently it had been shown that inorganic tin substances may react under certain conditions with the derivatisation reagent sodium tetraethylborate (G.3.8) to form organic tin compounds (especially methyl tin). This may lead to false-positive results for organic tin for example for coated tin toys where samples had been scraped off during sample preparation or wet oil paints stored in tinplate containers.

It is recommended to take special care in these circumstances to avoid false-positive results by

— repeating the scrape off process by avoiding the scrape off of metallic parts and/or

— using a further organic tin method (e.g. ISO/TS 16179) for confirming the EN 71-3 results.

This subject of possible false results will lead to an improved method on organic tins in a revised future version of EN 71-3. [A2]

H.11 pH value

[A1] After sample preparation a migration procedure under acidic conditions is performed. The concentration of the hydrochloric acid which is added and the pH control are parameters of relevance for the purpose of this migration procedure. Therefore, it is important that the hydrochloric acid

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solutions used in the extraction step are carefully prepared and standardized before use. Due to the instability of diluted hydrochloric acid solutions, checks should be done regularly.

Experience has shown that deviations in the pH value during the migration procedure can have a significant impact on the results of the analysis. Therefore, it is important to exactly follow the specified procedure and the subclauses on specific materials.

After adding the hydrochloric acid it can take several minutes until a stable pH value is reached. In most cases the pH value will be stable. In some special cases the pH value may change throughout the migration and the effects of this are being investigated in the framework of the revision of this European Standard.

It is important that the equipment specified in 6.2.2 is capable of measuring pH with accuracy required by this standard. In practice a pH meter with an accuracy of ± 0,02 is sufficient when calibrated in acidic conditions (pH 1,68 [12]). A1

A1

H.12 De-waxing (see 7.3.2)

Every sample needs to be assessed if a de-waxing procedure given under 7.3.2 is required. In case of a doubt, a comparison of the results of the untreated material with the de-waxed material needs to be assessed. Examples for such an evaluation are leads for colour pencils. A1

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Annex I
(informative)

Conformity assessment

I.1 General

This standard contains requirements and test methods for the migration of certain elements in a wide range of toys and toy materials. The compliance of toys with the element migration requirements of Directive 2009/48/EC [1] can be demonstrated by compliance with this standard. It should be noted however that final product testing and certification may not be the most appropriate and effective means to achieve this. Other means are available to economic operators;

— Verification of conformity of toy materials using supplier's declarations and technical specifications with the assessment of that information; supported by the appropriate documentation:

  Directive 2009/48/EC [1] contains requirements for the migration of some elements which may only be used in certain types of materials. According to the explanatory guidance document [3], manufacturers may assess the likelihood of toy materials to contain a prohibited or restricted substance. The scope of any testing in accordance with this standard may be based on the outcome of that assessment. On this basis, any actual testing can be aimed only at those substances that can be expected to appear in the toy material in question. Records of the assessment should be maintained within the technical documentation for the toy in question

— Other means with equivalent results:

  The use of alternative test methods may also be useful to show compliance with the requirements of this standard. The determination of elemental tin in migration solutions may be used to infer compliance with the requirements of organic tin when both inorganic and organic tin are available in the same migration solution. If the total content of an element in a toy material is known, and less than a maximum migration limit, this can be used to demonstrate compliance.

I.2 Chromium VI

As a first action method, the migration of total Chromium can be determined (see E on general elements). If the migration of total Chromium (i.e. Chromium (III) + Chromium (VI)) is below the maximum limit for Chromium (VI), it can be inferred that the material complies with the requirements for both Chromium (III) and Chromium (VI).

For Chromium VI the method in Annex F the limit of quantification is higher than the migration limit for category I and II toy materials. Compliance with the limits cannot be demonstrated by a migration test, however other options are available:

— determination of the content of total Chromium;

— safety assessment of the *toy material*.

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Annex J
(informative)

List of other organic tin compounds

In Table J.1 an overview is given of other organic tin compounds that may be present in toy materials. This is based on their occurrence in food contact materials or their (expected) registration. The method in Annex G has not been tested or validated for these compounds.

A1

Table J.1 — Overview other organic tin compounds

Organic tin compound	CAS-Nr	family
2-ethylhexyl 4,4-dibutyl-10-ethyl-7-oxo-8- oxa-3,5-dithia-4-stannatetradecanoate	10584–98–2	DBT
Dibutyltin diacetate	1067–33–0	DBT
2-ethylhexyl 10-ethyl-4,4-dioctyl-7-oxo-8-oxa-3,5-dithia-4-stannatetrade	15571–58–1	DOT
Dimethyltin bis(isooctylmercaptoacetate)	26636–01–1	DMT
Di-n-octyltin bis(ethylmaleate)	68109–88–6	DOT
2-ethylhexyl 10-ethyl-4-[[2-[(2-ethylhexyl) oxy]-2-oxoethyl]thio]-4-octyl-7-oxo-8-ox a-3,5-dithia-4-stannatetradecanoate	27107–89–7	MOT
Monomethyltin tris(2-ehma)	57583–34–3	MeT
Dimethyltin bis(2-ehma)	57583–35–4	DMT
Dibutyltin dilaurate	77–58–7	DBT
Dibutyltin maleate	78–04–6	DBT
Di-n-butyltin-bis(methylmaleinate)	15546–11–9	DBT

A1

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Annex ZA
(informative)

Relationship between this European Standard and the Essential Requirements of EU Directive 2009/48/EC aimed to be covered

This European Standard has been prepared under a Commission's standardization request Mandate M/445 to provide one voluntary means of conforming to essential requirements of Directive 2009/48/EC.

Once this standard is cited in the Official Journal of the European Union under that Directive, compliance with the normative clauses of this standard given in Table ZA.1 confers, within the limits of the scope of this standard, a presumption of conformity with the corresponding essential requirements of that Directive, and associated EFTA regulations.

A3

Table ZA.1 — Correspondence between this European Standard and Directive 2009/48/EC

Clause(s)/subclause(s) of this EN	Essential Requirements (ERs) of Directive 2009/48/EC	Qualifying remarks/Notes
4.2 Specific requirements	Annex II, III point 13	The limits for cadmium were amended by Commission Directive 2012/7/EU. The limits for barium were amended by Commission Regulation (EU) No 681/2013. The limits for lead were amended by Council Directive (EU) 2017/738.

A3

WARNING 1 — Presumption of conformity stays valid only as long as a reference to this European Standard is maintained in the list published in the Official Journal of the European Union. Users of this standard should consult frequently the latest list published in the Official Journal of the European Union.

WARNING 2 — Other Union legislation may be applicable to the product(s) falling within the scope of this standard.

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[2] European Commission Guidance document No. 12 on the application of the Directive on the safety of toys – packaging

[3] [A1] The European Commission, *An explanatory guidance document (to Directive 2009/48/EC)* [A1]

[4] Commission Directive 2012/7/EU of 2 March 2012 amending, for the purpose of adaptation to technical progress, part III of Annex II to Directive 2009/48/EC of the European Parliament and of the Council relating to toy safety

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[14] [A3] Commission Regulation (EU) No 681/2013 of 17 July 2013 amending part III of Annex II to Directive 2009/48/EC of the European Parliament and of the Council on the safety of toys

[15] Council Directive (EU) 2017/738 of 27 March 2017 amending, for the purpose of adapting to technical progress, Annex II to Directive 2009/48/EC of the European Parliament and of the Council on the safety of toys, as regards lead". [A3]

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