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English version

Safety of toys — Part 3: Migration of certain elements

Securite des jouets —
Partie 3: Migration de certains elements

Sicherheit von Spielzeug —
Teil 3: Migration bestimmter Elemente

This European Standard was approved by CEN on 1994-12-13. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Foreword

This European Standard was prepared by CEN/TC 52, Safety of toys, of which the secretariat is held by DS.

This European Standard has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EC Directive(s).

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of an identical text or by endorsement, at the latest by June 1995, and conflicting national standards shall be withdrawn at the latest by June 1995.

This standard constitutes the third Part of the European Standard on *Safety of toys*.

This Part should be read in conjunction with Part 1.

This standard specifies requirements and test methods for the migration of the elements antimony, arsenic, barium, cadmium, chromium, lead, mercury and selenium from toy material.

This standard contains 4 annexes.

- Annex A (normative) Test method to determine acidity of 1,1,1-trichloroethane
- Annex B (normative) Sieve requirements
- Annex C (informative) Preparation and analysis of test portions
- Annex D (informative) Background and rationale for the requirements and test methods

This standard is the result of the revision of EN 71-3 : 1988.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

Introduction

This European Standard for 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 6: *Graphical symbol for age warning labelling*

This standard is Part 3 of the European Standard on safety of toys, EN 71.

The requirements of this standard are based on bioavailability resulting from the use of toys and should not as an objective exceed the below mentioned levels per day:

- 0,2 µg for antimony;
- 0,1 µg for arsenic;
- 25,0 µg for barium;
- 0,6 µg for cadmium;
- 0,3 µg for chromium;
- 0,7 µg for lead;
- 0,5 µg for mercury;
- 5,0 µg for selenium;

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

Combining the daily intake with the bioavailability figures listed above, limits are obtained for various toxic elements in microgram per gram (milligram per kilogram) and are detailed in table 1. The figures obtained have been adjusted to minimize childrens' exposure to toxic elements and to ensure analytical feasibility taking into account limits achievable under current manufacturing conditions. (See annex D.)

1 Scope

This Part of this European Standard specifies requirements and test methods for the migration of the elements antimony, arsenic, barium, cadmium, chromium, lead, mercury and selenium from toy materials and from parts of toys except materials not accessible (see Part 1 of this standard).

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

When appropriate, the toy is subjected to relevant tests, specified in Part 1 of this standard, before the accessibility is considered.

Requirements are included for the migration from the following toy materials:

- coatings of paints, varnishes, lacquers, printing inks, polymers and similar coatings (see 8.1);
- polymeric and similar materials, including laminates, whether textile reinforced or not, but excluding other textiles (see 8.2);
- paper and paper board (see 8.3);
- textiles, whether natural or synthetic (see 8.4);
- glass/ceramic/metallic materials (see 8.5);
- other materials whether mass coloured or not (e.g. wood, fibre board, hard board, bone and leather) (see 8.6);
- materials intended to leave a trace (e.g. the graphite materials in pencils and liquid ink in pens) (see 8.7);
- pliable modelling materials, including modelling clays, and gels (see 8.8);
- paints, including finger paints, varnishes, lacquers, glazing powders and similar materials in solid or in liquid form appearing as such in the toy (see 8.9).

Toys and parts of toys which, due to their accessibility, function, mass, size or other characteristics, obviously exclude any hazard due to sucking, licking or swallowing, bearing in mind the normal and foreseeable behaviour of children, are not covered by this Part of EN 71.

NOTE. For the purposes of this standard, the following criteria are considered appropriate in the categorization of sucking, licking or swallowing:

- all intended food/oral contact toys, cosmetic toys and writing instruments categorized as toys.
- toys intended for children up to 6 years of age, i.e. all accessible parts and components where there is a probability that those parts or components may come into contact with the mouth. (See annex D.)

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

- EN 71-1 *Safety of toys — Part 1: Mechanical and physical properties*
- ISO 3696 *Water for analytical laboratory use — Specification and test methods*

3 Definitions

For the purposes of this standard the following definitions apply:

3.1 base material

Material upon which coatings may be formed or deposited.

3.2 coating

All layers of material formed or deposited on the base material or toy and includes paints, varnishes, lacquers, inks, polymers or other substances of a similar nature, whether they contain metallic particles or not, of a similar nature no matter how it has been applied to the toy and which can be removed by scraping with a sharp blade.

3.3 detection limit of a method

Three times the standard deviation of the blank value.

3.4 other materials, whether mass coloured or not

Materials such as wood, leather and other porous substances which may absorb colouring matter without forming a coating.

3.5 paper and paper board

A maximum mass per unit area of 400 g/m² is the limit for treating material under this category. Above this mass per unit area the substance is treated as other materials, such as wood, fibre board or hard board, etc.

3.6 scraping

Mechanical removal of coatings down to the base material.

3.7 toy material

All the accessible materials present in toys.

Element		Sb	As	Ba	Cd	Cr	Pb	Hg	Se
Maximum migrated element in mg/kg toy material	Any toy material given in clause 1 except for: - modelling clay - finger paint	60	25	1000	75	60	90	60	500
	Modelling clay and finger paint	60	25	250	50	25	90	25	500

4 Requirements

4.1 Specific requirements

The migration of elements from toys and parts of toys as specified in clause 1 shall comply with the limits given in table 1 when tested in accordance with clauses 7, 8 and 9. This requirement does not apply to lead solder when used for electric connections. (See annex D.)

4.2 Interpretation of results

The analytical result of materials established in clauses 7, 8 and 9 shall be adjusted by subtracting the analytical correction in table 2 to obtain an adjusted analytical result.

Materials are deemed to comply with the requirements of this standard if the adjusted analytical result is less than or equal to the limits in table 1. (See annex D.)

NOTE. Due to the precision of the methods specified in this standard an adjusted analytical result is required to take into consideration the results of interlaboratory trials. (See annex D.)

Example: Analytical result of lead 120 mg/kg
Analytical correction from table 2: 30 %

$$\text{Adjusted analytical result} = 120 - \frac{120 \times 30}{100} = 120 - 36$$

Adjusted analytical result = 84 mg/kg. This is deemed as complying with the requirements of the standard. (Lead 90 mg/kg.)

5 Principle

Soluble elements are extracted from toy materials under the conditions which stimulate the material remaining in contact with stomach acid for a period of time after swallowing. The concentrations of the soluble elements are determined quantitatively.

6 Reagents and apparatus

NOTE. No recommendation is made for the reagents, materials, and apparatus necessary for carrying out the analytical tests specified in clause 9.

6.1 Reagents

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

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

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

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

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

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

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

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

6.2 Apparatus

Normal laboratory apparatus and

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

6.2.2 *A means of measuring pH*, with an accuracy of $\pm 0,2$ pH units. Cross-contamination shall be prevented. (See annex D.)

6.2.3 *Membrane filter*, with a pore size of 0,45 μm .

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

6.2.4 *Centrifuge*, capable of centrifuging at $(5000 \pm 500) g^1$. (See annex D.)

6.2.5 *A means to agitate the mixture*, at a temperature of $(37 \pm 2)^\circ C$.

6.2.6 *A selection of containers of gross volume*, between 1,6 times and 5,0 times that of the volume of hydrochloric acid extractant.

7 Selection of test portions

A laboratory sample for testing shall consist of a toy either in the form in which it is marketed, or in the form in which it is intended to be marketed. Test portions shall be taken from accessible parts (see EN 71-1) of a single toy sample, i.e. identical materials in the toy may be combined and treated as a single test portion but additional toy samples shall not be used. Test portions are only permitted to be composed of more than one material or colour where physical separation, e.g. dot printing, patterned textiles or mass limitation reasons, precludes the formation of discrete specimens. (See annex D.)

NOTE. The requirement does not preclude that test portions can be taken from materials in a form such that they are representative of the relevant material specified above and the substrate upon which they are deposited. (See annex D.)

Test portions where less than 10 mg of material are available are not tested.

8 Preparation and analysis of test portions

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

8.1.1 *Sample removal/preparation procedure*

Remove the coating from the laboratory sample by mechanical means at room temperature and comminute it at a temperature not exceeding ambient. Obtain a test portion of not less than 100 mg passing through a metal sieve of aperture 0,5 mm (see 6.2.1).

Where there is only between 10 mg and 100 mg of comminuted uniform coating available this shall be tested in accordance with 8.1.2 and the quantity of the appropriate elements shall be calculated as if 100 mg of test portion had been used and the mass of the test portion shall be reported under 10e.

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

8.1.2 *Test procedure*

Using the appropriate sized container (see 6.2.6), mix the test portion so prepared with 50 times its mass of an aqueous solution at $(37 \pm 2)^\circ C$ of $c(HCl) = 0,07 \text{ mol/l}$ (see 6.1.1). 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 at $(37 \pm 2)^\circ C$. Shake for 1 min. Check the acidity of the mixture. If the pH is greater than 1,5 add dropwise, while shaking the mixture, an aqueous solution of $c(HCl)$ approximately 2 mol/l (see 6.1.4) until the pH is between 1,0 and 1,5. Protect the mixture from light. Agitate the mixture at $(37 \pm 2)^\circ C$ (see 6.2.5) for 1 h continuously and then allow to stand for 1 h at $(37 \pm 2)^\circ C$.

Without delay, efficiently separate the solids from the solution, firstly by filtration using a membrane filter (see 6.2.3), and if necessary, by centrifuging at up to $5000 g^1$ (see 6.2.4). Separation shall be completed as soon as possible after the completion of the standing time; centrifuging shall take no longer than 10 min and shall be reported under 10e.

If the resulting solutions are to be kept for more than the working day prior to analysis they shall be stabilized by addition of hydrochloric acid so that the concentration of the stored solution is approximately $c(HCl) = 1 \text{ mol/l}$.

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

8.2.1 *Sample removal/preparation procedure*

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

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

If the laboratory sample is not uniform in its material, a test portion shall be obtained from each different material forming a mass greater than 10 mg. In the case where there is between 10 mg and 100 mg of uniform material the mass of the test portion shall be reported under 10e and the quantity of the appropriate elements shall be calculated as if 100 mg of the test portion had been used.

8.2.2 *Test procedure*

Follow the procedure in 8.1.2.

¹⁾ $g = 9,80665 \text{ m/s}^2$.

8.3 Paper and paper board

8.3.1 Sample removal/preparation procedure

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

If the laboratory sample is not uniform in its material, a test portion shall be obtained from each different material forming a mass of not less than 100 mg. Where there is between 10 mg and 100 mg of uniform material the mass of the test portion shall be reported under 10e and the quantity of the appropriate elements shall be calculated as if 100 mg of the test portion had been used.

If the paper or paper board to be tested is coated with a coating of paint, varnish, lacquer, printing ink, adhesive or similar coating, test portions of the coating shall not be taken separately. In such cases test portions shall be taken from the material in accordance with this subclause in a way that they also include representative parts of the coated area. Test portions so obtained shall be taken in accordance with relevant methods specified in this subclause and this shall be reported under 10e. (See annex D.)

8.3.2 Test procedure

Macerate the test portion so prepared with 25 times its mass of water (see 6.1.7) at $(37 \pm 2)^\circ\text{C}$ so that the resulting mixture is homogeneous. Quantitatively transfer the mixture to the appropriate sized container (see 6.2.6). Add to the mixture an aqueous solution of $c(\text{HCl}) = 0,14 \text{ mol/l}$ (see 6.1.2) at $(37 \pm 2)^\circ\text{C}$ at 25 times the mass of the test portion.

Shake for 1 min. Check the acidity of the mixture. If the pH is greater than 1,5 add dropwise, while shaking the mixture, an aqueous solution of $c(\text{HCl})$ approximately 2 mol/l (see 6.1.4) until the pH is between 1,0 and 1,5. Protect the mixture from light. Agitate the mixture at $(37 \pm 2)^\circ\text{C}$ (see 6.2.5) for 1 h continuously and then allow to stand for 1 h at $(37 \pm 2)^\circ\text{C}$.

Without delay, efficiently separate the solids from the solution, firstly by filtration using a membrane filter (see 6.2.3), and if necessary, by centrifuging at up to $5000 g^{1)}$ (see 6.2.4). Separation shall be completed as soon as possible after the completion of the standing time; centrifuging shall take no longer than 10 min and shall be reported under 10e.

If the resulting solutions are to be kept for more than the working day prior to analysis they shall be stabilized by addition of hydrochloric acid so that the concentration of the stored solution is approximately $c(\text{HCl}) = 1 \text{ mol/l}$.

8.4 Textiles, whether natural or synthetic

8.4.1 Sample removal/preparation procedure

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

If the sample is not uniform in its material or colour, a test portion shall be obtained from each different material forming a mass greater than 100 mg. A portion forming a mass between 10 mg and 100 mg shall form part of the test portion obtained from the main material.

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

8.4.2 Test procedure

Follow the procedure in 8.1.2.

8.5 Glass/ceramic/metallic materials

8.5.1 Sample removal/preparation procedure

Toys and components shall be first subjected to the relevant tests in accordance with EN 71-1. If the toy or component fits entirely within the small parts cylinder and contains accessible glass, ceramic or metallic materials then the toy shall be tested in accordance with 8.5.2 after removal of any coating in accordance with 8.1.1. (See annex D.)

NOTE. Toys and components that have no accessible glass, ceramic or metallic materials are not tested according to 8.5.2. (See annex D.)

8.5.2 Test procedure

Place the toy or component in a 50 ml glass container with nominal dimensions: height 60 mm, diameter 40 mm. Add a sufficient volume of an aqueous solution of $c(\text{HCl}) = 0,07 \text{ mol/l}$ (see 6.1.1) at $(37 \pm 2)^\circ\text{C}$ to just cover the toy or component. Cover the container, protect the contents from light and allow the contents to stand for 2 h at $(37 \pm 2)^\circ\text{C}$.

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

Without delay, efficiently separate the solids from the solution, firstly by decantation followed by filtration using a membrane filter (see 6.2.3), and if necessary, by centrifuging at up to $5000 g^{1)}$ (see 6.2.4). Separation shall be completed as soon as possible after the completion of the standing time; centrifuging shall take no longer than 10 min and shall be reported under 10e.

If the resulting solutions are to be kept for more than 24 h prior to analysis they shall be stabilized by addition of hydrochloric acid so that the concentration of the stored solution is approximately $c(\text{HCl}) = 1 \text{ mol/l}$.

¹⁾ $g = 9,80665 \text{ m/s}^2$.

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

8.6.1 Sample removal/preparation procedures

Obtain a test portion of not less than 100 mg of the material according to 8.2.1, 8.3.1, 8.4.1 or 8.5.1 whichever is appropriate.

If the laboratory sample is not uniform in its material, a test portion shall be obtained from each different material forming a mass greater than 10 mg and 100 mg of uniform material the mass of the test portion shall be reported under 10e and the quantity of the appropriate elements shall be calculated as if 100 mg of the test portion had been used.

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

8.6.2 Test procedures

The materials shall be tested by the most appropriate method of 8.2.2, 8.3.2, 8.4.2 or 8.5.2. The method used shall be reported under 10e.

8.7 Materials intended to leave a trace

8.7.1 Sample removal/preparation procedure for materials in solid form

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

A test portion shall be obtained from each different material intended to leave a trace, present in the laboratory sample, and forming a mass greater than 10 mg. Where there is between 10 mg and 100 mg of material, the mass of the test portion shall be reported under 10e and the quantity of the appropriate elements shall be calculated as if 100 mg of the test portion had been used. If the material contains any grease, oil, wax or similar material, the test portion shall be enclosed in hardened filter-paper and these ingredients shall be removed with

1,1,1-trichloroethane or other suitable solvent by using solvent-extraction (see 6.1.6). Analytical measures shall be taken to ensure that removal of the ingredients referred to is quantitative. The solvent used shall be reported under 10e.

8.7.2 Sample removal/preparation procedure for materials in liquid form

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

A test portion shall be obtained from each different material intended to leave a trace, present in the laboratory sample, and forming a mass greater than 10 mg. Where there is between 10 mg

and 100 mg of material, the mass of the test portion shall be reported under 10e and the quantity of the appropriate elements shall be calculated as if 100 mg of the test portion had been used. If the material is intended to solidify in normal use and contains grease, oil, wax or similar material, the test portion shall be allowed to solidify under normal use conditions and the resulting material shall be enclosed in hardened filter-paper and the grease, oil, wax or similar material shall be removed with 1,1,1-trichloroethane or other suitable solvent (6.1.6) by using solvent-extraction. Analytical measures shall be taken to ensure that removal of the ingredients referred to is quantitative. The solvent used shall be reported under 10e.

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

Using the appropriate sized container (see 6.2.6), mix the test portion so prepared with 50 times its mass of an aqueous solution at $(37 \pm 2)^\circ\text{C}$ of $c(\text{HCl}) = 0,07 \text{ mol/l}$ (see 6.1.1). For a test portion mass of between 10 mg and 100 mg, mix the test portion with 5,0 ml of this solution at $(37 \pm 2)^\circ\text{C}$. Shake for 1 min. Check the acidity of the mixture.

If the test portion contains large quantities of alkaline materials, generally in the form of calcium carbonate, adjust the pH to between 1,0 and 1,5 with hydrochloric acid $c(\text{HCl})$ approximately 6 mol/l (see 6.1.5) in order to avoid overdilution. The amount of hydrochloric acid used in relation to the amount of solution shall be reported under 10e. If only small quantities of alkaline material are present and the pH of the mixture is greater than 1,5 add dropwise, while shaking the mixture, an aqueous solution of $c(\text{HCl})$ approximately 2 mol/l (see 6.1.4) until the pH is between 1,0 and 1,5. Protect the mixture from light. Agitate the mixture at $(37 \pm 2)^\circ\text{C}$ (see 6.2.5) for 1 h continuously and then allow to stand for 1 h at $(37 \pm 2)^\circ\text{C}$.

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

With the test portion remaining in the hardened filter-paper, macerate the test portion so prepared with 25 times the mass of the original material with water (see 6.1.7) at $(37 \pm 2)^\circ\text{C}$ so that the resulting mixture is homogeneous. Quantitatively transfer the mixture to the appropriate sized container (see 6.2.6). Add to the mixture an aqueous solution of $c(\text{HCl}) = 0,14 \text{ mol/l}$ (see 6.1.2) at $(37 \pm 2)^\circ\text{C}$ in the proportion of 25 times the mass of the original test portion. In the case of a test portion mass between 10 mg and 100 mg macerate the test portion with 2,5 ml of water (see 6.1.7). Quantitatively transfer the mixture to the appropriate sized container (see 6.2.6). Add 2,5 ml of $c(\text{HCl}) = 0,14 \text{ mol/l}$ (see 6.1.2) at $(37 \pm 2)^\circ\text{C}$ to the mixture. Shake for 1 min. Check

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

If only a small quantity of alkaline material is present and the pH of the mixture is greater than 1,5 add dropwise, while shaking the mixture, an aqueous solution of $c(\text{HCl})$ approximately 2 mol/l (see 6.1.4) until the pH is between 1,0 and 1,5. Protect the mixture from light. Agitate the mixture at $(37 \pm 2)^\circ\text{C}$ (see 6.2.5) for 1 h continuously and then allow to stand for 1 h at $(37 \pm 2)^\circ\text{C}$.

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

Without delay, efficiently separate the solids from the solution, firstly by filtration using a membrane filter (see 6.2.3), and if necessary, by centrifuging at up to 5000 g^1 (see 6.2.4). Separation shall be completed as soon as possible after the completion of the standing time: centrifuging shall take no longer than 10 min and shall be reported under 10e.

If the resulting solutions are to be kept for more than the working day prior to analysis they shall be stabilized by addition of hydrochloric acid so that the concentration of the stored solution is approximately $c(\text{HCl}) = 1 \text{ mol/l}$.

8.8 Pliable modelling materials, including modelling clays, and gels

8.8.1 Sample removal/preparation procedure

Obtain a test portion of not less than 100 mg of the material from the laboratory sample.

A test portion shall be obtained from each different material in the laboratory sample. If the material contains grease, oil, wax or similar material, the test portion shall be enclosed in hardened filter-paper and these ingredients shall be removed with 1,1,1-trichloroethane or other suitable solvent by using solvent-extraction (see 6.1.6). Analytical measures shall be taken to ensure that removal of the ingredients referred to is quantitative. The solvent used shall be reported under 10e.

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

Using the appropriate sized container (see 6.2.6) mix the test portion so prepared after breaking up of clay or doughy materials, if appropriate, with 50 times its mass of an aqueous solution at $(37 \pm 2)^\circ\text{C}$ of $c(\text{HCl}) = 0,07 \text{ mol/l}$ (see 6.1.1).

Shake the mixture for 1 min. Check the acidity of the mixture. If the test portion contains large quantities of alkaline materials, generally in the form of calcium carbonate, adjust the pH to between 1,0 and 1,5 with hydrochloric acid $c(\text{HCl})$ approximately 6 mol/l (see 6.1.5) in order to avoid overdilution. The amount of hydrochloric acid used in relation to the amount of solution shall be reported under 10e. If only a small quantity of alkaline material is present and the pH of the mixture is greater than 1,5 add dropwise, while shaking the mixture, an aqueous solution of $c(\text{HCl})$ approximately 2 mol/l (see 6.1.4) until the pH is between 1,0 and 1,5. Protect the mixture from light. Agitate the mixture at $(37 \pm 2)^\circ\text{C}$ (see 6.2.5) for 1 h continuously and then allow to stand for 1 h at $(37 \pm 2)^\circ\text{C}$.

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

With the test portion remaining in the hardened filter-paper, macerate the test portion so prepared with 25 times the mass of the original material with water (see 6.1.7) at $(37 \pm 2)^\circ\text{C}$ so that the resulting mixture is homogeneous. Quantitatively transfer the mixture to the appropriate sized container (see 6.2.6). Add to the mixture an aqueous solution of $c(\text{HCl}) = 0,14 \text{ mol/l}$ (see 6.1.2) at $(37 \pm 2)^\circ\text{C}$ in the proportion of 25 times the mass of the original test portion. Shake for 1 min. Check the acidity of the mixture. If the test portion contains large quantities of alkaline materials, generally in the form of calcium carbonate, adjust the pH between 1,0 and 1,5 with $c(\text{HCl})$ approximately 6 mol/l (see 6.1.5) in order to avoid overdilution. The amount of hydrochloric acid used in relation to the amount of solution shall be reported under 10e.

If only a small quantity of alkaline material is present and the pH of the mixture is greater than 1,5 add dropwise, while shaking the mixture, an aqueous solution of $c(\text{HCl})$ approximately 2,0 mol/l (see 6.1.4) until the pH is between 1,0 and 1,5. Protect the mixture from light. Agitate the mixture at $(37 \pm 2)^\circ\text{C}$ (see 6.2.5) for 1 h continuously and then allow to stand for 1 h at $(37 \pm 2)^\circ\text{C}$.

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

Without delay, efficiently separate the solids from the solution, firstly by filtration using a membrane filter (see 6.2.3), and if necessary, by centrifuging at up to 5000 g^1 (see 6.2.4). Separation shall be completed as soon as possible after the completion of the standing time; centrifuging shall take no longer than 10 min and shall be reported under 10e.

If the resulting solutions are to be kept for more than the working day prior to analysis they shall be stabilized by addition of hydrochloric acid so that the concentration of the stored solution is approximately $c(\text{HCl}) = 1 \text{ mol/l}$.

¹⁾ $\text{g} = 9,80665 \text{ m/s}^2$.

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

8.9.1 Sample removal/preparation procedure for materials in solid form

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

A test portion shall be obtained from each different material, present in the laboratory sample, and forming a mass greater than 10 mg. Where there is between 10 mg and 100 mg of material, the mass of the test portion shall be reported under 10e and the quantity of the appropriate elements shall be calculated as if 100 mg of the test portion had been used. If the material contains any grease, oil, wax or similar material the test portion shall be enclosed in hardened filter-paper and these ingredients shall be removed with

1,1,1-trichloroethane or other suitable solvent by using solvent-extraction (see 6.1.6). Analytical measures shall be taken to ensure that removal of the ingredients referred to is quantitative. The solvent used shall be reported under 10e.

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

8.9.2 Sample removal/preparation procedure for materials in liquid form

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

A test portion shall be obtained from each different material, present in the laboratory sample, and forming a mass greater than 10 mg. In the case where there is between 10 mg and 100 mg of material, the mass of the test portion shall be reported under 10e and the quantity of the appropriate elements shall be calculated as if 100 mg of the test portion had been used. If the material is intended to solidify in normal use and contains grease, oil, wax or similar material, the test portion shall be allowed to solidify under normal use conditions and the resulting material shall be enclosed in hardened filter-paper and the grease, oil, wax or similar material shall be removed with 1,1,1-trichloroethane or other suitable solvent using solvent-extraction (see 6.1.6). Analytical measures shall be taken to ensure that removal of the ingredients referred to is quantitative. The solvent used shall be reported under 10e.

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

Follow the procedure in 8.7.3.

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

With the test portion remaining in the filter-paper, macerate the test portion so prepared with 25 times the mass of the original material with water (see 6.1.7) at $(37 \pm 2)^\circ\text{C}$ so that the resulting mixture is homogeneous. Quantitatively transfer the mixture to the appropriate sized container (see 6.2.6). Add to the mixture an aqueous solution of $c(\text{HCl}) = 0,14 \text{ mol/l}$ (see 6.1.2) at $(37 \pm 2)^\circ\text{C}$ in the proportion of 25 times the mass of the original test portion. Shake for 1 min. Check the acidity of the mixture. If the test portion contains large quantities of alkaline materials, generally in the form of calcium carbonate, adjust the pH to between 1,0 and 1,5 with $c(\text{HCl})$ approximately 6 mol/l (see 6.1.5) in order to avoid overdilution. The amount of hydrochloric acid used in relation to the amount of solution shall be reported under 10e.

If only a small quantity of alkaline material is present and the pH of the mixture is greater than 1,5 add dropwise, while shaking the mixture, an aqueous solution of $c(\text{HCl})$ approximately 2 mol/l (see 6.1.4) until the pH is between 1,0 and 1,5. Protect the mixture from light. Agitate the mixture $(37 \pm 2)^\circ\text{C}$ (see 6.2.5) for 1 h continuously and then allow to stand for 1 h at $(37 \pm 2)^\circ\text{C}$.

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

Without delay, efficiently separate the solids from the solution, firstly by filtration using a membrane filter (see 6.2.3), and if necessary, by centrifuging at up to 5000 g^1 (see 6.2.4). Separation shall be completed as soon as possible after the completion of the standing time; centrifuging shall take no longer than 10 min and shall be reported under 10e.

If the resulting solutions are to be kept for more than one working day prior to analysis they shall be stabilized by addition of hydrochloric acid so that the concentration of the stored solution is approximately $c(\text{HCl}) = 1 \text{ mol/l}$.

¹⁾ $g = 9,80665 \text{ m/s}^2$

9 Determination of the quantity of migrated elements

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

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

10 Test report

The test report shall contain at least the following information:

- a) type and identification of the product and/or material tested;
- b) a reference to this European Standard (EN 71-3 : 1994);
- c) the techniques used for determining the quantity of each element and the detection limit if the limit is deviating from the requirements in clause 9;
- d) the results of the tests expressed as mg element/kg material, stating that the result is related to the soluble element;
- e) details of the procedure used (from clause 8) to prepare the test portion (including for example, if base material was incorporated, if centrifuging was required to separate the solids from the solution prior to analysis, if additional acid was required to lower the pH, if the ratio of solid to acid extractant exceeds 1 : 50 and the solvent used for removing any grease, oil, wax or similar ingredient in toy materials) and if the specimen was adjusted to 1 mol/l for overnight storage;
- f) any departure by agreement or otherwise from the test procedure specified;
- g) date of the test.

Annex A (normative)

Test method to determine acidity of 1,1,1-trichloroethane

A.1 Reagents

A.1.1 *Standard sodium hydroxide*,
(0,1 ± 0,005) mol/l.

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

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

A.2 Test method

Place 100 cm³ of neutralized water in a 250 cm³
flask, add 100 cm³ of 1,1,1-trichloroethane sample,
stopper the flask and shake vigorously. Allow the
layers to separate, add 0,5 cm³ of phenolphthalein
indicator and titrate with 0,1 mol/l aqueous sodium
hydroxide using a microburette until the upper
layer turns a faint pink colour.

A.3 Calculation

The acidity in parts per million by mass of
hydrochloric acid is calculated from the following
equation.

$$\text{Acidity} = \frac{36,5}{d} T_1$$

T_1 is the volume in cm³ of 0,1 mol/l sodium
hydroxide;

d is the relative density of the sample of
1,1,1-trichloroethane.

Annex B (normative)
Sieve requirements

Table B.1 Sieve dimensions and tolerances				
Dimensions in millimetres				
Nominal aperture size	Nominal wire diameter in test sieve	Tolerances		
		Maximum deviation for size of an individual aperture	Tolerance for average aperture	Intermediate deviation (no more than 6 % of the apertures to exceed the nominal plus this figure)
0,500	0,315	+ 0,090	$\pm 0,018$	+ 0,054

Annex C (informative)

Preparation and analysis of test portions

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

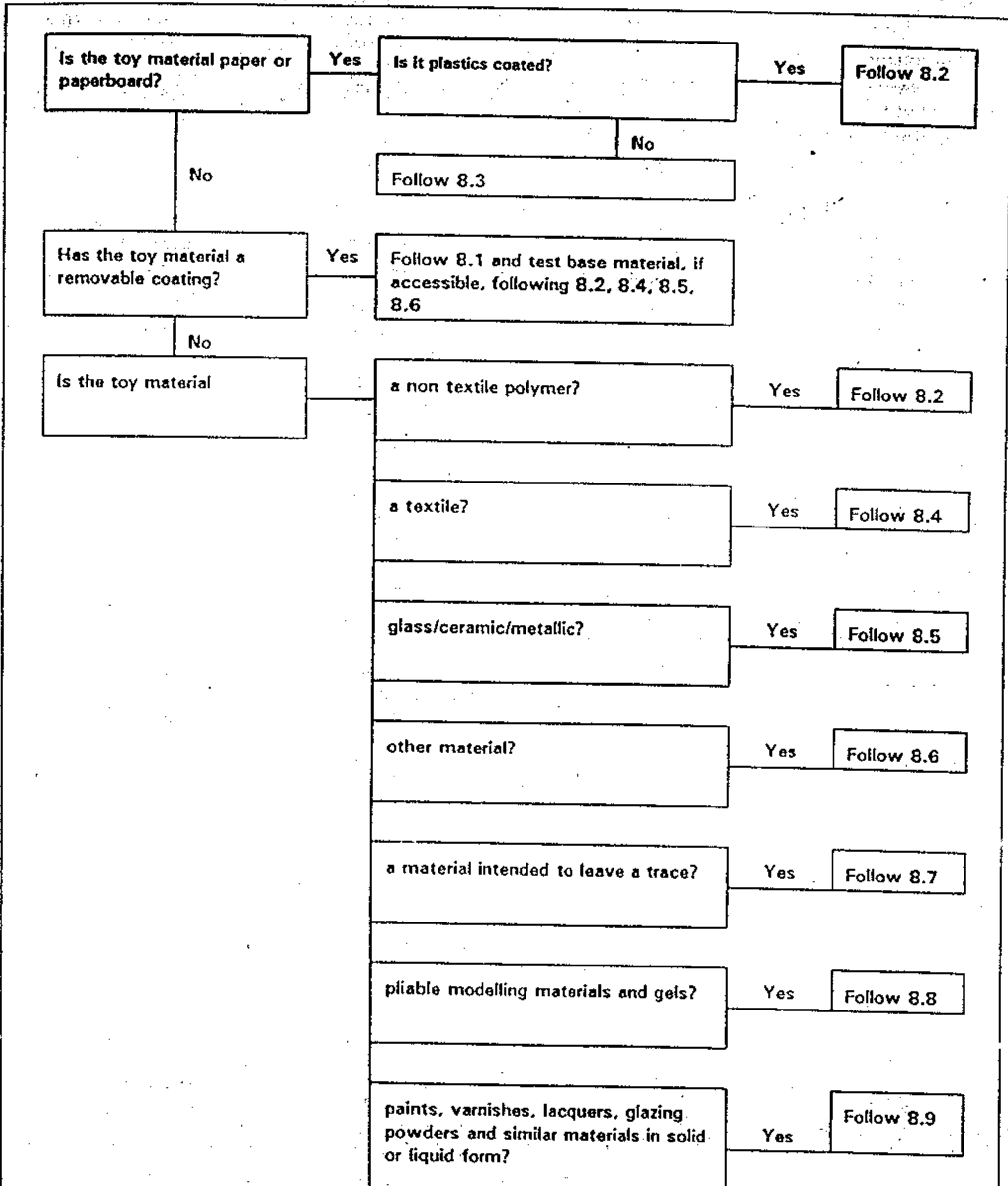


Figure C.1 Diagram showing the procedure and analysis of test portions

Annex D (informative)

Background and rationale for the requirements and test methods in this Part of EN 71

D.1 Introduction

The way bioavailability is defined in the Toy Safety Directive²⁾ led to the test methods in the standard addressing the amount of soluble element migration from a toy material.

The approach of total element determinations was discounted because of the following reasons.

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

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

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

D.2 Scope

D.2.1 Packaging

'Unless part of the toy' is suggested to mean, for example, boxes containing jigsaw puzzles or where the instructions are included on the packaging in the case of games, etc., but taking into account the 2nd indent of the note in the scope restricting the requirements to toys intended for children up to 6 years of age. It is not intended to address for example, blister packs containing simple instructions.

D.2.2 Scope note

The note is intended to indicate an approach to the decision of what toys, or toy components are excluded from the standard because of characteristics that render them unlikely to present a risk of injury by the ingestion of materials containing the toxic elements.

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

- as children get older there is less of a tendency for them to place toy/toy materials in their mouths and therefore the risk presented by the ingestion of toxic elements is reduced;
- the larger the toy, or the less accessible the material, the risk of ingestion of toxic elements is diminished.

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

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

D.3 Specific requirements (see 4.1)

The limit of soluble barium has been raised from 500 mg/kg to 1000 mg/kg for the following reasons.

The use of barium sulfate in toys has led to levels of soluble barium in the extraction solution $c(\text{HCl}) = (0,07 \pm 0,005) \text{ mol/l}$ at 37°C of between 400 mg/kg and 600 mg/kg as expressed in the weight of toy material taken. This level is such that due to the statistical uncertainty of the determination, a PASS or FAIL could not be indicated.

The formation of non-bioavailable colloidal barium sulfate crystals in the filtrate resulting in levels of apparently soluble barium which exceed 500 mg/kg because of problems with filtration.

In addition, the previous limit of migration of barium of 500 mg/kg from toy materials was not consistent with 25,0 µg/day bioavailability and the 8 mg/day of toy material intake; 25,0 µg corresponds to a migration limit of 3,125 g/kg. It is understood that the 500 mg/kg limit was consciously selected despite the 'theoretical' 3,125 g/kg figure. The effect of the 500 mg/kg limit was to reduce the bioavailability from the advised 25 µg to 4 µg. It should be noted that the 25,0 µg figure has been reduced from an initial 50,0 µg, not for toxicological reasons, but according to the Commission of the European Communities, 'to reduce the avoidable input on body burden'.

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

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

²⁾ See European Council Directive 88/378/EEC of 3 May 1988 concerning the safety of toys (published in the official journal of the EC No. L 187 of 16 July 1988).

Because of the way bioavailability is defined in the Toy Safety Directive²⁾, the test methods in the standard measure the amount of soluble element migration from a toy material. With this type of chemical test the result is dependent upon the specified conditions of test and there is no absolute or true value. Consequently, it is more difficult to obtain close statistical agreement between laboratories when performing such migration tests.

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

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

There is no direct relationship between the total element content of a toy material and the soluble migration of that element under the standard test conditions. Therefore, measuring the total element content and converting the result to give a soluble element figure is not an answer to this problem. Setting maximum total element limits is a possibility but requires an amendment to the Toy Safety Directive²⁾. (See also D.1.)

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

As a result, a defined scraping and comminution procedure for collecting a 300 µm to 500 µm paint test portion was proposed as a revision to the test procedure. A European interlaboratory trial was conducted in 1993 with 29 participating laboratories to compare the amended procedure with that in the 1988 standard.

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

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

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

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

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

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

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

²⁾ See page 14.

D.5 Reagents (see 6.1)

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

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

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

D.6 Apparatus (see 6.2)

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

See D.4.

D.6.2 A means of measuring pH (see 6.2.2)

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

D.6.3 Centrifuge (see 6.2.4)

This is a new subclause and specifies the performance requirements for centrifuging limits and amount of time permitted for centrifuging (to 10 min) and requires this to be reported under 10e. The latter is necessary as centrifuging has been reported to increase the extraction of barium.

D.6.4 A selection of containers (see 6.2.6)

The indication of the gross volume of the containers is intended to ensure adequate movement of the solution leading to a more efficient extraction.

D.7 Selection of test portions (see clause 7)

The practice of analysing 'composite' (combination of different materials or colours) test portions is neither appropriate nor will it normally be necessary (with the availability of the '5,0 ml' test method). The analysis of composite materials is not satisfactory because from a theoretical viewpoint it can result in the reduction in the migration of toxic elements that would otherwise occur. A simple example is known to have occurred, i.e. barium extraction from paint was reduced when co-extracted with another paint. This may have been the result of a counter-ion in the second paint which would cause the barium to be precipitated. Sulfate is such a counter-ion; whether sulfate itself was involved is not known and is not important for the establishment of this principle. Thus, except for the cases where the separation of colours or toy material is impractical, e.g. dot printing, each discrete area is treated as a single sample.

The note makes it possible to test toy materials which are not in the form of a toy for reference purposes. However, the standard clearly requires taking of test portions from the toy itself.

D.8 Paper and paper board (see 8.3)

D.8.1 Sample removal/preparation procedure (see 8.3.1)

Paper and paper board are to be treated as if they were a single material, i.e. surface coatings, if present, are not to be removed, but test portions will include representative parts of the surface. This procedure has been adopted because in the practical situation of a child chewing paper and paper board, preferential removal of a coating is unlikely and the substrate is equally important.

D.9 Textiles, whether natural or synthetic (see 8.4)

D.9.1 Sample removal/preparation procedure (see 8.4.1)

It is not feasible to take separate coloured test portions from a complex patterned fabric. It is therefore required that a single test portion be taken that represents all the colours in the material.

D.10 Glass/ceramic/metallic materials (see 8.5)

D.10.1 Sample removal/preparation procedure (see 8.5.1)

Toys or components which do not fit entirely within the 'small parts cylinder' of EN 71-1 are not tested because there is no hazard from ingestion and no significant extraction occurs with saliva simulator. The small parts cylinder is used to assess the size of toy/toy components of all relevant age groups. Comminution of glass, ceramic and metallic materials is inappropriate. Agitation of the test solution would be impractical for many examples and thus extraction is carried out without shaking. The diameter of the vessel and the orientation of the test portion have been selected to minimize variables.

Glass, ceramic and metallic materials completely coated so that no glass, ceramic or metal is accessible as defined in EN 71-1 are not tested according to this requirement.

Where glass, ceramic and metal surfaces are accessible even when partially covered by a coating these are tested in accordance with 8.5.2 after removal of the partial coating entirely according to the method in 8.1.1. This procedure is a compromise as only a single toy may be taken as a sample as specified in clause 7 of this standard.

D.11 Other materials, whether mass coloured or not (see 8.6)

This subclause incorporates mass coloured materials and other materials which are not coloured such as wood, hardboard, leather and bone, etc. which may have received some other treatment, but were not covered by the 1988 standard.