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Determination of total concentration of certain elements in toys

玩具中特定元素总含量的测定

(ISO 8124-5:2015, Safety of toys - Part 5: Determination of total concentration of certain elements in toys, MOD)

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WARNING - The personnel using this Standard shall have practical experience in regular laboratory work. This Standard does not point out all possible safety problems. It is the responsibility of the user to take appropriate safety and health measures and to ensure compliance with the conditions of relevant national regulations.

1 Scope

1.1 This Standard specifies the sampling, digestion and instrumental analysis methods for the determination of the total concentration of certain elements of antimony, arsenic, barium, cadmium, chromium, lead, mercury and selenium in toy materials and toy parts.

Note: Other certain elements, after verification, can be determined with reference to the test methods of this Standard. Manufacturers are encouraged to apply the test methods of this Standard and the limits from GB 6675.4-2014 to raw materials used in the manufacture of toys to give increased certainty of conformity to the requirements of GB 6675.4-2014.

1.2 This Standard applies to the following types of toy materials:

- coatings of paints, varnishes, lacquers, printing inks, polymers, and similar coatings;
- polymeric and similar materials, including laminates, whether textile-reinforced or not, but excluding other textiles;
- paper, paperboard, and cardboard;
- natural or synthetic textiles;
- metallic materials whether coated or not;
- other dip dyeable materials, whether mass-coloured or not (e.g. wood, fibreboard, hardboard, bone, and leather);
- materials intended to leave a trace (e.g. the graphite materials in pencils and liquid ink in pens);
- pliable modelling materials, including modelling clays and gels;

metallic particles or not, no matter how they have been applied to the toy.

Note: This definition includes metallic coatings deposited on a metal surface such as an electroplated coating. However, electroplating will only require testing if it can be removed by **scraping** (3.6); otherwise, it may be tested with the base material.

3.3

Laboratory sample

Product either in the form in which it is marketed, or in the form in which it is intended to be marketed.

3.4

Test portion

Single material taken from an accessible part of a **laboratory sample** (3.3).

3.5

Composite test portion

Test portion that is composed of more than one similar material type.

Note: This definition excludes the mixing of different types of materials, such as not allowing the mixing of textiles and paint coatings.

3.6

Scraping

Mechanical process for removal of **coatings** (3.2) down to the **base material** (3.1).

3.7

Complete digestion

Complete breakdown of the test portion.

3.8

Sample blank

Solution that has undergone the same digestion processes used for the digestion of **test portions** (3.4) and consists of all reagents excluding the test portion.

5.2.2 Inductively coupled plasma mass spectrometer (ICP-MS).

5.2.3 Inductively coupled plasma emission spectrometer (ICP-OES).

5.2.4 Microwave digestion system, equipped with sample holder and digestion vessel. The digestion vessel is a closed vessel suitable for microwave digestion. It is advisable to use a digestion vessel that can withstand a temperature of at least 225 °C and an internal pressure of at least 3000 kPa. The liner of the vessel shall be PTFE (polytetrafluoroethylene)/TFM [*tris*-(α-trifluoromethyl-β,β-difluorovinyl)-1,3,5-enzenetricarboxylate], or PTFE/PFA (perfluoroalkoxy ethylene) or another chemically inert material. Vessels shall also be equipped with a safety relief valve or disc that will prevent vessel rupture or ejection of the vessel cap.

Note 1: Some new microwave digestion systems do not need to use high-pressure digestion vessels. As long as they can achieve the same digestion effect, these systems can also be used.

Note 2: For microwave digestion systems of different models and different manufacturers used in different laboratories, there may be many different and specific safety guidelines and operating recommendations. Operators need to, according to the equipment manual, and the safety operation guidelines provided by the manufacturer or related to the literature, correctly operate and use microwave digestion equipment and digestion vessel (see A.3, precautions for microwave digestion).

Note 3: The inner liners shall be inspected regularly to check for any chemical or physical degradation.

Note 4: Internal pressures in excess of 3000 kPa can occur with some samples, e.g. crayons, and so a suitable pressure-rated vessel, e.g. 5000 kPa, should be used in these cases.

5.2.5 Electric hot plate. The surface temperature can reach at least 140 °C.

Note: Provided that the hot plate is capable of handling the extra heating required, use of a 12 mm to 25 mm thick heat-resistant glass plate placed on the hot plate can help reduce the presence of hot spots common to electric hot plates.

5.2.6 Graphite digester, heatable sleeve device with temperature adjustment function (equipment with programmed setting or temperature rising function can be selected). Heating temperature can reach at least 140 °C. It is equipped with a suitable digestion container.

5.2.7 Scalpel, or other suitable scraping or cutting tools.

7 Preparation of test portions

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

At room temperature, the coating is obtained from the toy sample by scraping; take care to avoid scraping the base material. Where lithographic coatings (dot printing) are present, it is impractical to separate the individual colours and so select a representative test portion. Under the condition of not exceeding the ambient temperature, the sample is divided into small pieces with a length of no more than 2 mm in any direction, to facilitate the digestion.

Some coatings deposited on non-polymeric base material are more difficult to scratch. It is permissible to add a few drops of solvent, such as methylene chloride (5.1.6) or acetone/ethanol solution (5.1.7), to soften the coating and facilitate scraping.

If a solvent treatment is used, it shall make sure that all solvents remaining in the sample are completely removed by evaporation before weighing.

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

Scrape-off, cut, or grind the clean, dry material into pieces having a maximum length in any dimension of 2 mm using a scalpel or other suitable scraping or cutting tool.

7.3 Paper, paperboard and cardboard

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

If the paper or paperboard to be tested is coated with paint, varnish, lacquer, printing ink, adhesive, or similar coating, test portions of the coating shall not be taken separately. In such cases, take test portions from the material so that they also include representative parts of the coated area. Material that is printed, where the ink has become part of the base material, is prepared as though they are unprinted.

7.4 Natural or synthetic textiles

Cut the material into pieces having a maximum length in any dimension of 2 mm using a suitable cutting tool. The material or colour with a mass of 10 mg~100 mg shall be taken as part of the test portion obtained from the main material; shall be tested separately as a test portion different from the main tested in the dry or liquid state.

7.9 Metallic materials whether or not coated

First, according to GB 6675.2, carry out small parts test on toys or components. If the toy or toy component can be completely accommodated in small parts instrument and contains accessible metallic materials, the metallic materials shall be tested.

Using suitable cutting and grinding tools, obtain a test portion of between 10 mg and 100 mg.

Where practical, obtain the test portion from an uncoated part of the metallic material. If the metal part is coated, remove any coatings (including electroplated coatings) that can be scraped off. Electroplating that cannot be removed by scraping shall be tested together with the metallic base material. Materials less than 10 mg are exempt from testing (see 6.1, selection of test portions), and are indicated in the report [see Clause 12 c)].

8 Digestion and analysis of test portions

8.1 General

Clause 8 provides three digestion methods: microwave digestion (8.2), electric hot plate and graphite digester digestion (8.3). Digestion of samples with electric hot plate and graphite digester shall be carried out in a fume hood. 8.2 (microwave digestion) and 8.3 (electric hot plate and graphite digester digestion) describe that, how to use aqua regia [3 parts of hydrochloric acid (5.1.4) and 1 part of nitric acid (5.1.2)] and reverse aqua regia [1 part of hydrochloric acid (5.1.4) and 3 parts of nitric acid (5.1.2)] to digest the test portions. Then, use inductively coupled plasma mass spectrometer (ICP-MS) or inductively coupled plasma emission spectrometer (ICP-OES) for quantitative analysis. Other instruments whose detection limits meet the requirements (see Table 1) can also be used.

Aqua regia and reverse aqua regia are best prepared in situ in the presence of the test portion. The order of mixing the hydrochloric acid and nitric acid is not critical but should be the same for consistency. Sample blanks are pretreated with each batch of samples.

Allow the laboratory to use appropriate different microwave digestion systems and digestion conditions. However, the laboratory shall, based on the equipment manufacturer's recommendations and experience in the digestion of different types of materials, determine the best digestion conditions. The most basic requirement is complete digestion. If the safety relief valve or safety

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

If the digestion is incomplete (e.g. evidence of original sample, solid lumps of charred test portion), the test should be repeated using a new test portion and alternative digestion conditions until a complete digestion has been achieved. In the event that a complete digestion cannot be achieved, an alternative method must be used (see A.4.2, incomplete digestion). Such methods are outside the scope of this Standard and their use shall be indicated in the final test report [see Clause 12 d)].

When the digestion is complete, quantitatively transfer the digestate with washings to a beaker and evaporate to about 1 mL on a hot plate. Allow to cool and then add about 4 mL to 5 mL of water followed by 3 to 4 drops of concentrated hydrochloric acid (5.1.4). Filter the digest solution into a 25 mL volumetric flask (5.2.13) or where limited sample was available, into a 10 mL volumetric flask (5.2.13), to avoid excessive dilution. Dilute to the mark with water (5.1.1). This diluted digest shall be subjected to instrumental quantification as soon as practically possible.

8.3 Digestion with electric hot plate and graphite digester

8.3.1 Analysis by ICP-OES

Weigh 10 mg~100 mg of test portion, accurate to 0.1 mg; place it in a 25 mL beaker or other digestion vessel; carefully add 4.5 mL of concentrated hydrochloric acid (5.1.4); and then add 1.5 mL of concentrated nitric acid (5.1.2). After the reaction is complete, add dropwise 1 mL of hydrogen peroxide (5.1.5) (optional). If the reaction vessel is a beaker, it shall be covered with a watch glass. Heat on the electric hot plate (5.2.5) or graphite digester (5.2.6) (surface temperature is about 140 °C), until most of the acid is evaporated.

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

If brown fumes are still observed after digestion for 1 h or particles are observed in the solution, continue the heating. Add an appropriate amount of concentrated hydrochloric acid (5.1.4) and concentrated nitric acid (5.1.2) in the same ratio (3:1) to prevent the sample from becoming dry. When the sample

Annex A

(Informative)

Background and rationale

A.1 Use and applicability

A.1.1 General

This Standard is intended to provide a means of determining whether a certain element is present in a toy material and at what total concentration. This results may be used to decide whether further testing to GB 6675.4-2014 is required. For example in the case that all elements of concern have a total concentration at or below the levels set out in GB 6675.4-2014, Table 1 it would be impossible for the material to show a migration level in excess of the limits in GB 6675.4-2014, Table 1.

Note: This Standard only addresses the 8 elements listed in Table 1 of GB 6675.4-2014. The test laboratory shall conduct internal verification of the methods detailed in this Standard. The total concentration result can also be used to determine whether it violates other regulations or standards on the total concentration of certain elements.

As stated in 1.2, this Standard is not applicable to glass, ceramic, and siliceous materials. Methods for the determination of total concentration of certain elements in these materials can be found in other standards such as CPSC-CH-E1002-08.3.

A.1.2 Compositing of test portions

Composite testing is feasible, but care must be taken in order to avoid an error of the analytical results. If very small sample masses are used in composite tests, errors in weighing can become significant. Therefore, whenever possible, the mass of the composite test portion should be around 100 mg. In cases where this is not practical, the mass of an individual test portion is not permitted to be less than 10 mg. A sufficient amount of material has to be used for each material in the composite test portion, giving proper consideration for the weighing capabilities of the balance used, the detection limits following the dilution of the digested solution (see Table 1) and for the subsequent instrumental analysis. The compositing of different types of materials is not permitted, e.g. compositing textiles and paint coatings. Only similar materials can be grouped together into a composite test portion (3.5). When calculating the concentration of a target element in a material, it is assumed that all of that element found in the digested sample originated from just one of the composite

ρ_E - the element mass concentration measured by the instrument, in milligrams per liter (mg/L);

V_f - constant volume, in milliliters (mL);

m_1 - mass of a single test portion, in grams (g).

In the example above, it can be seen that the composite test portion result of 825 mg/kg marginally exceeds the safety limit; but by using the exact mass of the individual test portion, a more precise decision on re-testing can be made on a material by material basis. In the example, the yellow and red plastics results both exceed the safety limit of 800 mg/kg and so should be re-tested as individual materials. The blue plastic is below the safety limit and so need not be re-tested.

A.2 Scope

The wording “that form part of the toy” in 1.2 is intended to mean, for example, boxes containing jigsaw puzzles or packaging on which the instructions are included in the case of games, etc. It is not intended to address packs containing only simple instructions.

A.3 Precautions relating to microwave digestion

This Standard does not specify the use conditions of microwave digestion system. This allows the test laboratory to use an appropriate microwave system and digestion conditions. However, the test laboratory should determine the optimum digestion conditions based upon the equipment manufacturer‘s recommendations and the laboratory’s own experiences with digestion of various material types. It is essential that complete digestion of test portion is achieved.

Microwave digestion equipment varies from manufacturer to manufacturer. Always consult the specific equipment manual and manufacturer’s literature for proper and safe operation of the microwave equipment and vessels. Some precautions are common to most designs and are noted below:

- Inspect the inner liners of the microwave digestion vessel regularly to check for any chemical or physical degradation and replace any defective parts.
- The digestion methods involve the use of strong acids which are corrosive and cause burns. Laboratory coats, gloves, and safety glasses should be worn when handling acids.
- Toxic and irritating fumes are released by nitric acid and hydrochloric acid. Always carry out digestion in a fume cupboard and when adding acid to

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Contact: Wayne Zheng, Sales@ChineseStandard.net

Linkin: https://www.linkedin.com/in/waynezhengwenrui/

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