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**[Including 2026XG1] Determination of certain phthalate esters  
in toys and children products**

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### 1 Scope

This document describes the determination methods for the plasticizer requirements in

5.3.7 of GB 6675.1-2014.

This document describes the determination methods for 7 kinds of phthalate esters (according to the specifications in Table A.1) in toys and children's products, i.e., di-iso-butyl phthalate (DIBP), di-n-butyl phthalate (DBP), benzyl butyl phthalate (BBP), bis-(2-ethylhexyl) phthalate (DEHP), di-n-octyl phthalate (DNOP), di-iso-nonyl phthalate (DINP) and di-iso-decyl phthalate (DIDP).

This document is applicable to toys and children's products containing polymers and similar

materials, textiles, coatings and liquid materials, and applicable to 5.3.7 of GB 6675.1-2014. This document has been validated for polyvinyl chloride (PVC), polyurethane (PU) and some representative paint coatings, see Annex B. Other materials for toys and children's products can be determined with reference to this document after validation.

The test of other phthalate esters can refer to this document after validation.

## 2 Normative references

The contents of the following documents constitute the essential provisions of this document through normative references in the text. Among them, for dated references, only the version corresponding to the date applies to this document; for undated references, the latest version (including all amendments) applies to this document.

GB/T 454 Paper - Determination of bursting strength (GB/T 454-2020, ISO 2758:2014, MOD)

GB 6675.1-2014 Toys safety - Part 1: Basic code

GB 6675.2-2014 Safety of toys - Part 2: Mechanical and physical properties (ISO 8124-1:2000, MOD)

GB 6675.4-2014 Safety of toys - Part 4: Migration of certain elements (ISO 8124-3:2010, MOD)

prepare a series of standard working solutions (prepare at least 5 standard working solutions, of which the mass concentration of DIBP, DBP, BBP, DEHP, DNOP is 0.4 mg/L ~ 10 mg/L, and the mass concentration of DINP, DIDP is 2 mg/L ~ 50 mg/L), each of the working solutions contains 10 mg/L of the internal standard.

The mass concentration of the internal standard can be adjusted according to the mass concentration of the test portion, but the mass concentration of the internal standard in the final sample solution (8.3.3) shall be consistent with the mass concentration of the internal standard in the standard working solution. The standard working solution containing the internal standard should be stored at a temperature of 0 °C ~ 8 °C, and the validity period is 1 month.

## 6 Instruments and equipment

6.1 For normal laboratory glassware, phthalate esters are common contaminants, and even a small amount can affect quantitative results. It is recommended to avoid the use of any plastic equipment that may contaminate the analysis. Glassware and equipment should be cleaned before use.

6.2 Gas chromatography/mass spectrometry (GC-MS): equipped with a quartz capillary column and a mass spectrometer detector (EI source).

6.3 Soxhlet extractor: see Figure C.1. When necessary, with a cooling circulation device.

6.4 Solvent extractor (also known as fat extractor), see Figure C.2. When necessary, with a cooling circulation device.

6.5 Extraction thimble: made of cellulose.

6.6 Cotton wool.

6.7 Analytical balance: an accuracy of 1 mg.

6.8 Concentration apparatus: such as a rotary evaporator, etc.

6.9 Solid phase extraction cartridge (SPE): 1 g silica gel/6 mL tubes, or equivalent.

6.10 Volumetric flasks: of 5 mL, 10 mL, 25 mL, 50 mL and 100 mL capacity.

6.11 Pipettes: of 0.2 mL, 0.5 mL, 1 mL, 2 mL, 5 mL and 10 mL capacity.

6.12 Organic phase microporous membrane filter: of pore size 0.45  $\mu\text{m}$ , recommended to use polytetrafluoroethylene (PTFE) microporous membrane filter.

6.13 Ultrasonic extractor: thermostatically controlled internally or externally, with the effective ultrasonic power density ranging from 0.25 W/cm<sup>2</sup> to 2.0 W/cm<sup>2</sup>.

Example:

One of the following methods can be selected for extraction according to the actual situation.

8.2.1 Method A Place the test portion in the extraction thimble (6.5) of a 250 mL Soxhlet extractor (6.3).

In order to prevent the test portion from floating, cotton wool (6.6) can be placed on the upper layer of the extraction thimble.

Add 120 mL of dichloromethane (5.1) into a 250 mL round-bottom flask, extract for 6 h, and reflux at least 4 times per hour. If necessary, use a cooling circulation device for cooling and refluxing.

The volume of the dichloromethane can be adjusted according to the extraction equipment.

After cooling, use a suitable concentration apparatus (6.8) to concentrate until about 10 mL of the extract remains, and take care to prevent it from being completely evaporated to dryness.

It is recommended to control the temperature of the water bath of a rotary evaporator at 40 °C ~ 50 °C, while maintaining the pressure at 30 kPa ~ 45 kPa.

Care shall be taken to control the temperature of reflux and concentration to prevent the loss of phthalate esters.

8.2.2 Method B Place the test portion in the extraction thimble (6.5) of a solvent extractor (6.4). In order to prevent the test portion from floating, cotton wool (6.6) can be placed on the upper layer of the extraction paper tube.

Add 80 mL of dichloromethane (5.1) into the extraction bottle, set a suitable temperature (such as 80 °C), make the dichloromethane boil and reflux, extract for 1.5 h, then rinse for 1.5 h; if necessary, use a cooling circulation device for cooling and refluxing. Finally, concentrate the extract to about 10 mL.

The volume of the dichloromethane can be adjusted according to the extraction equipment.

Care shall be taken to control the temperature of reflux and concentration to prevent the loss of phthalate esters.

### 8.2.3 Method C

8.2.3.1 Solid materials Place the test portion in an airtight glass reaction vessel (6.15), and add 25 mL of dichloromethane. Place the container in an ultrasonic extractor (6.13), the liquid level in the ultrasonic tank shall be higher than the liquid level of the dichloromethane in the reaction vessel, start at  $(60 \pm 2)$  °C, and continue ultrasonication for 60 min. The ultrasonic extractor performance check shall be carried out according to Annex E.

NOTE: If the material does not dissolve or swell in dichloromethane, Method A or Method B might be preferable.

8.2.3.2 Liquid materials Place the test portion in an airtight glass reaction vessel (6.15), and add 15 mL of dichloromethane. Place the container in the ultrasonic extractor (6.13), the liquid level in the ultrasonic water tank shall be higher than the liquid level of the dichloromethane in the reaction vessel, start at  $(60 \pm 2)$  °C, and continue ultrasonication for 60 min. The ultrasonic extractor performance check shall be carried out according to Annex E.

## 8.3 Preparation of analytical solutions

8.3.1 General If the extract obtained in 8.2.1, 8.2.2 or 8.2.3 is turbid, it can be centrifuged with a centrifuge (6.16) at a centrifugal force of 5000 g before filtration. If necessary, the extract can be purified with a solid phase extraction cartridge (6.9). Before purification, pre-wash and activate with 10 mL of dichloromethane, discard the activation solution, and then load the test portion, rinse with 3 mL of dichloromethane for 3 times and collect the eluate.

The extract or the eluent, at room temperature, can be made up to the mark by 8.3.2 external standard method or 8.3.3 internal standard method according to the actual situation.

### 8.3.2 Constant volume (external standard method)

8.3.2.1 Method A and Method B Transfer the extract or the eluent into a 25 mL volumetric