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**Determination of nine primary aromatic amines in toys - Gas
chromatography-mass spectrometry**

玩具中9种初级芳香胺含量的测定 气相色谱-质谱联用法

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Foreword

This document was drafted in accordance with the rules given in GB/T 1.1-2020 "Directives for standardization - Part 1: Rules for the structure and drafting of standardizing documents".

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This document was proposed by China Light Industry Federation.

This document shall be under the jurisdiction of National Technical Committee on Toys of Standardization Administration of China (SAC/TC 253).

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Determination of nine primary aromatic amines in toys -- Gas chromatography-mass spectrometry WARNING -- The personnel using this document shall have practical experience working in a formal laboratory. This document does not indicate all possible security issues. Users are responsible for taking appropriate safety and health measures and ensuring compliance with relevant national regulations.

1 Scope

This document describes the gas chromatography-mass spectrometry method for the determination of nine primary aromatic amines in toy materials, including aniline, ortho toluidine, 2-methoxyaniline, 4-chloroaniline, 2-naphthylamine, benzidine, 3,3'-dimethylbenzidine, 3,3' - dichlorobenzidine, and 3,3' - dimethoxybenzidine.

This document applies to toy parts and materials such as textiles, leather, wood, paper, liquids, flexible molding materials, etc. that contain coloring agents (including dyes and pigments).

This document is not applicable to finger painting pigments.

2 Normative references

GB/T 6682

3 Terms and definitions

There are no terms or definitions that need to be defined in this document.

4 Principle

Extract the specimen by ultrasonic extraction with an appropriate solvent. Conduct purification and concentration through a diatomaceous earth column. Set it to a set volume. Use gas chromatography-mass spectrometry for determination. Qualify based on its retention time and characteristic ions. Quantify with external standard method.

5 Reagent and materials

5.1 Water. GB/T 6682, Grade three water.

5.2 Methanol. chromatographically pure.

5.3 Tert butyl methyl ether. chromatographically pure.

5.4 9 primary aromatic amine standard substances/standard samples. according to the requirements of Annex A, the purity is greater than 98%.

5.5 Standard stock solution (1000 µg/mL or higher concentration). prepare the above

standard substance/sample (5.4) into a standard stock solution using methanol (5.2) or other suitable solvents. Accurately weigh to 0.1 mg.

NOTE. Standard stock solution is stored in brown bottles. Store at -18°C. It shall be valid for 3 months.

5.6 Mixed standard working solution. dilute the standard reserve solution (5.5) step by

step with methanol (5.2) or other suitable solvents to prepare a series of mixed standard working solutions with mass concentrations of 0.2 µg/mL, 0.5 µg/mL, 1.0 µg/mL, 2.5 µg/mL, and 5.0 µg/mL, respectively.

NOTE. As needed, it can be prepared to other suitable concentrations.

5.7 Diatomaceous earth. Porous granular, burned at 600°C for 4 h. After cooling, store in a dryer for later use.

6 Instruments

6.1 Gas chromatography-mass spectrometer (equipped with EI ionization source).

6.2 Analytical balance. precision is 0.1 mg.

6.3 Vortex oscillator. speed range is from 0 r/min~3000 r/min.

6.4 Ultrasonic extractor. capable of internal or external temperature control. The effective power density range of ultrasound is 0.25 W/cm²~2.0 W/cm².

Example. For an ultrasonic extractor with a total power of 1200 W (including 200 W ultrasonic effective power and 1000 W heating power), the water bath area is 400 cm², and the ultrasonic effective power density is 0.50 W/cm² (=200 W/400 cm²).

6.5 Centrifuge. centrifugal force is (5000 ± 500) g.

6.6 Diatomaceous earth column. a glass or polypropylene column with an inner

diameter of 25 mm~30 mm and a length of 130 mm~150 mm. Fill approximately 20 g

NOTE. Excessive flow rate during elution can lead to incomplete extraction of aromatic amines, resulting in low recovery rates.

8.1.2 Clay and crystal clay specimens

Weigh 1 g of the specimen to the nearest 0.1 mg. Place it in a 50 mL centrifuge tube.

First, add 2 mL~3 mL of methanol (5.2) and mix thoroughly. Add a certain volume of water to make the total volume 15 mL. Vortex in a vortex oscillator (6.3) for 30 s. After mixing, use an ultrasonic extractor (6.4) to extract for 15 min at room temperature.

When necessary, centrifuge for 5 min using a centrifuge (6.5) with a centrifugal force of (5000 ± 500) g. Transfer all the supernatant into a diatomaceous earth column (6.6).

Adsorb for 20 min. Wash twice with tert butyl methyl ether, 40 mL each time. The flow rate shall be controlled to form droplets during elution. Collect tert butyl methyl ether eluent in a round bottom flask with a standard grinding mouth or a glass container suitable for vacuum rotary evaporator (6.7).

8.2 Concentration

Place the round bottom flask containing the elution solution of tert butyl methyl ether collected above on a vacuum rotary evaporator (6.7). Concentrate to about 5 mL under low vacuum conditions at (35 ± 2)°C. Then transfer to a nitrogen blower (6.8). Blow nitrogen to near dryness at room temperature (do not blow dry). Immediately and accurately transfer 1.0 mL of methanol (5.2) or other suitable solvent into a nearly dry round bottom flask. After vortex mixing, filter with a 0.45 µm filter membrane (6.9).

The obtained specimen solution shall be analyzed on the machine in a timely manner.

8.3 Blank test

Each batch of samples shall undergo a method blank test. Using the same testing procedure as the sample test, except without adding samples. The results of the blank test can be used to evaluate the contamination experienced during the testing process.

The blank test result shall be less than the quantification limit of the method (10.1).