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PEOPLE'S REPUBLIC OF CHINA**

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**Determination of N,N-dimethylformamide in toys and children's
products - Gas chromatography-mass spectrometry**

玩具及儿童用品材料中N,N-二甲基甲酰胺的测定 气相色谱-质谱法

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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".

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

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The drafting organizations of this document. Guangzhou Customs Technical Center, Huangpu Customs Technical Center, China Consumer Product Quality and Safety

Promotion Association, Shenzhen University of Technology, Zhejiang Qunying Vehicle Industry Co., Ltd., Luohe Rover Stationery Manufacturing Co., Ltd., Hangzhou Nomo Testing Technology Co., Ltd., Guangdong Research Institute of New Biomaterials and High-end Medical Devices, Yike Testing and Certification Co., Ltd., Nanjing Customs Light Industrial Products and Children's Products Testing Center, Aofei Entertainment Co., Ltd., Mingmen (China) Children's Products Co., Ltd., Shenzhen Metrology and Quality Inspection Institute, Shantou Customs Technical Center, Shanghai Customs Mechanical and Electrical Products Testing Technology Center, SGS-CSTC Standards Technical Services (Shanghai) Co., Ltd.

Main drafters of this document. Wu Shanshan, Lin Shaopeng, Guo Zongning, Fang Han, Du Hongzhong, Li Shidong, Huang Lina, Chu Junlong, Yan Jinghui, Lin Xianzhen, Huang Kanghui, Ji Changxin, Hong Jinqing, Lu Xuanben, Chen Zhigeng, Chen Qinxue, Lin Xiaowei, Huang Xuehong, Fu Rao, Zhang Xiaoning.

Determination of N, N-dimethylformamide in toys and children's products -- Gas chromatography-mass spectrometry

1 Scope

This document describes the determination of N, N-dimethylformamide in toys and children's product materials by gas chromatography-mass spectrometry.

This document is applicable to the determination of N, N-dimethylformamide content in toys and children's product materials.

2 Normative references

GB/T 22048-2022

3 Terms and definitions

There are no terms or definitions that require definition in this document.

4 Principle

Ethyl acetate is used as the extracting solution. Ultrasonic extraction is used to extract N, N-dimethylformamide from toys and children's products. The extract is filtered and then determined by gas chromatography-mass spectrometry. External standard method is used for quantification.

5 Reagents and materials

5.1 Ethyl acetate (CAS No. 141-78-6). chromatographically pure.

5.2 N, N-Dimethylformamide standard substance (CAS No. 68-12-2). purity $\geq 99\%$, or

with certified standard certificate, as per Annex A.

5.3 N, N-dimethylformamide standard stock solution. Accurately weigh an appropriate

amount of N, N-dimethylformamide standard substance (accurate to 0.1 mg). Use ethyl acetate (5.1) to prepare a standard stock solution with a mass concentration of 100 mg/L.

NOTE. The standard stock solution shall be stored at $0^{\circ}\text{C}\sim 8^{\circ}\text{C}$ away from light. The validity period is 1 month.

5.4 N, N-dimethylformamide intermediate standard solution. Accurately pipette an

appropriate amount of N, N-dimethylformamide standard stock solution (5.3). Use ethyl acetate (5.1) to prepare an intermediate standard solution with a mass concentration of 20 mg/L.

NOTE. The intermediate standard solution shall be stored at $0^{\circ}\text{C}\sim 8^{\circ}\text{C}$ away from light. The validity period is 1 month.

5.5 N, N-dimethylformamide standard working solution. Take appropriate amount of

N, N-dimethylformamide intermediate standard solution (5.4). Dilute with ethyl acetate (5.1) to prepare a series of standard working solutions with mass concentrations of 0.5 mg/L, 1 mg/L, 2 mg/L, 5 mg/L, and 10 mg/L respectively.

NOTE. The standard stock solution shall be stored at $0^{\circ}\text{C}\sim 8^{\circ}\text{C}$ away from light. The validity period is 1 month.

6 Instruments

6.1 Analytical balance. read to 0.1 mg.

6.2 Stoppered extraction bottle. 50 mL.

6.3 Ultrasonic extractor. with temperature control device; upper temperature limit

$\geq 60^{\circ}\text{C}$; the performance of ultrasonic extractor shall be in accordance with 6.13 of GB/T 22048-2022.

6.4 Organic filter membrane. pore size is $0.22\ \mu\text{m}$.

6.5 Gas chromatography-mass spectrometry (GC-MS). equipped with EI source.

7.1 Sample preparation and extraction

Obtain the single material from the sample by mechanical means. Use suitable tools to shape the sample into a size not exceeding 5 mm.

Use an analytical balance (6.1) to weigh 1 g (accurate to 0.1 mg) of sample. Place it in a stoppered extraction bottle (6.2). Add 20.0 mL of ethyl acetate solvent (5.1) and tighten the bottle cap. Place the sample in an ultrasonic extractor (6.3) in a 60°C water bath and ultrasonicate for 30 min. After cooling, filter the extract through an organic filter membrane (6.4). Use a gas chromatograph-mass spectrometer (6.5) to measure the extract).

7.2 Blank test

Except for not weighing the specimen, the remaining steps are the same as 7.1 to carry out a blank test.

7.3 Instrument analysis conditions

Since the test results depend on the instrument used, it is not possible to give universal parameters for chromatographic analysis. The parameters shall be set to ensure that the component being measured can be separated from other components to a limited extent during the determination. The following operating conditions have been proven to be suitable for the test.

- a) Capillary column. polar polyethylene glycol stationary phase, 30 m×0.25 mm×0.25 µm;
- b) Injection volume. 1 µL;
- c) Injection port temperature. 230°C;
- d) Chromatographic mass spectrometer interface temperature. 250°C;
- e) Transfer line temperature. 250°C;
- f) Column temperature. 50°C for 2 min; 20°C/min to 200°C, for 2 min;
- g) Injection system. non-divided flow;
- h) Carrier gas. helium;
- i) Carrier gas flow rate. 1.2 mL/min;
- j) Ionization mode. EI ionization;
- k) Ionization energy. 70 eV;
- l) Ion source temperature. 230°C;
- m) Solvent delay. 2 min;
- n) Scan mode. full scan mode and selected ion monitoring; mass scanning range is 30