NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB/T 24279.2-2021

Textiles - Determination of certain flame retardants - Part 2: Phosphorus flame retardants

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Foreword

This Part 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".

This Part is Part 2 of GB/T 24279 "Textiles - Determination of certain flame retardants". The following parts of GB/T 24279 have been issued:

- Part 1: Brominated flame retardants;
- Part 2: Phosphorus flame retardants.

This Part uses redrafting method to modify and adopt ISO 17881-2:2016 "Textiles - Determination of certain flame retardants - Part 2: Phosphorus flame retardants".

The structural differences between this Part and ISO 17881-2:2016 are as follows:

- added "Normative references" as Clause 2;

- added "Terms and definitions" as Clause 3;

- added "Precision" as Clause 9.

The technical differences between this Part and ISO 17881-2:2016 are as follows:

- In Clause 5, changed analytically pure to chromatographically pure, added the purity of the tested standard substance for liquid chromatography analysis; according to the relevant requirements of prohibited flame retardants, added the test substance "Di-(2,3-dibromopropyl) phosphate";

- in 5.8, added GB/T 6682 as a reference; specified the requirements for water used;

- in 6.7, added the balance with a division of 0.01g for weighing textile specimen;

- in Clause 8, added the provisions that dilution factors and calculation results are kept to one decimal place, so as to make the calculation and expression of results clearer;

- in Clause 9, according to relevant domestic standards, added relevant description on precision.

This Part made the following editorial modifications:

Textiles - Determination of certain flame retardants -

Part 2: Phosphorus flame retardants

WARNING - Personnel using this document should have practical experience in formal laboratory work. This document does not indicate all possible security issues. The user is responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated by relevant national laws and regulations.

1 Scope

This Part specifies the test method that uses the high-performance liquid chromatography-tandem mass spectrometer (HPLC-MS/MS) to determine

certain phosphorus flame retardants in textiles.

This Part is applicable to various textiles.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

GB/T 6682, Water for analytical laboratory use - Specification and test methods (GB/T 6682-2008, ISO 3696:1987, MOD)

3 Terms and definitions

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

4 Principle

Use acetone as extractant. Extract flame retardant from textile specimens in ultrasonic generator. Use HPLC-MS/MS to determine flame retardant. Use external standard method to quantify.

Use acetonitrile (5.6) as solvent to respectively prepare standard stock solution of each flame retardant (5.1~5.4). The concentration is 1000μg/mL.

7.1.2 Standard working solution

Use acetonitrile to prepare the mixed standard solution. According to the needs, use acetonitrile to dilute step by step into a series of mixed standard working solutions of suitable concentration. Choose at least five standard dilution solutions of appropriate concentration to establish the standard working curve. Conduct HPLC-MS/MS analysis.

NOTE: Store the standard stock solution in the dark at 0°C~4°C. The validity is 6 months. Store the standard working solution in the dark at 0°C~4°C. The validity is 3 months.

7.2 Specimen preparation

Take respective specimen from the sample. After cutting and mixing well, use the balance (6.7) to weigh (1.00+/-0.01)g of specimen.

7.3 Ultrasonic extraction

Place the specimen in a stoppered glass reaction flask (6.4). Add 20mL of acetone (5.5). Extract in 40°C ultrasonic generator (6.2) for 40min. Collect the extract in a 100mL concentrating bottle (6.5). Add another 20mL of acetone to the reaction flask. Extract 20min at about 40°C. Combine and collect the extracts in a 100mL concentrating flask (6.5).

Use a rotary evaporator (6.3) to concentrate until the residue is nearly dry. Take 2mL of acetonitrile (5.6) to dissolve the residue. Filter through organic filter membrane (6.6). The resulting filtrate is used for the determination of flame retardants.

7.4 Determination

Use HPLC-MS/MS(6.1) to determine the flame retardant in the filtrate obtained in 7.3. Annex A gives examples of detection parameters for HPLC-MS/MS determination of flame retardants. Carry out a blank test. Subtract the blank value.

When the flame retardant concentration is too low, appropriately increase the specimen mass so that the detection concentration is within the linear range of the instrument's response value.

When the flame retardant concentration is higher than the linear range of the instrument response value, the solution is appropriately diluted.

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