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Replacing GB/T 24168-2009

Determination of the content of phthalate in textile dyeing and finishing auxiliaries

纺织染整助剂产品中邻苯二甲酸酯的测定

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Foreword

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

This document replaces GB/T 24168-2009 "*Determination of the content of phthalate in textile dyeing and finishing auxiliaries*". Compared with GB/T 24168-2009, in addition to structural adjustments and editorial changes, the main technical changes are as follows:

- ADD the type of phthalate (see Clause 1 of this document);
- MODIFY the content of the reagents and materials (see 5.2 of this document, 4.2 of the 2009 edition);
- MODIFY the specifications of the extractor in the instruments and equipment [see 5.3.5 of this document, 4.3d) of the 2009 edition];
- ADD the requirements for analytical balance (see 5.3.4 of this document);
- MODIFY the gas chromatography-mass spectrometry analysis conditions (see 5.4 of this document, 4.4.2 of the 2009 edition);
- MODIFY the method for preparing standard solutions (see 5.5.1, 5.5.2, 5.5.3 of this document, 4.2 of the 2009 edition);
- MODIFY the method for preparing sample solutions (see 5.5.4 of this document, 4.4.1 of the 2009 edition);
- ADD the provisions for the maximum deviation of the relative ion abundance of characteristic ions in qualitative determination (see Table 2 of this document);
- MODIFY the detection limit and recovery rate (see 6.1, 6.2 of this document, 5.1, 5.2 of the 2009 edition);
- MODIFY the content of the test report (see Clause 7 of this document, Clause 6 of the 2009 edition).

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

This document was proposed by China Petroleum and Chemical Industry Federation.

This document shall be under the jurisdiction of National Technical Committee on Dyestuff of Standardization Administration of China (SAC/TC 134).

Determination of the content of phthalate in textile dyeing and finishing auxiliaries

WARNING -- People who use this document shall have practical experience in regular laboratory work. This document does not point out all possible safety issues. Users are responsible for taking appropriate safety and health measures and ensuring that the conditions stipulated by relevant national laws and regulations are met.

1 Scope

This document describes the gas chromatography-mass spectrometry (GC/MS) determination method for 24 kinds of phthalates in textile dyeing and finishing auxiliaries.

This document applies to the determination of phthalates in various categories of textile dyeing and finishing auxiliaries.

NOTE: The names of the 24 kinds of phthalates are shown in Annex A.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6682-2008 Water for analytical laboratory use - Specification and test methods

GB/T 8170-2008 Rules of rounding off for numerical values & expression and judgement of limiting values

3 Terms and definitions

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

4 Principle

After extraction with *n*-hexane-acetone mixed solvent or other suitable solvent, the textile dyeing and finishing auxiliary samples are determined by gas chromatography-mass spectrometry, the retention time and characteristic ions are used for qualitative

analysis, and the external standard method of the peak area of quantitative ions is used for quantitative analysis.

5 Determination method

5.1 General provisions

Unless otherwise specified, only reagents confirmed as analytical reagent and grade 3 water specified in GB/T 6682-2008 are used. The test results are determined according to the rounded value comparison method in 4.3.3 of GB/T 8170-2008.

5.2 Reagents and materials

5.2.1 Phthalate standard substances: meet the requirements of Annex A, and the content is ≥ 98 % (mass fraction).

5.2.2 *n*-hexane: chromatographically pure.

5.2.3 Acetone: chromatographically pure.

5.2.4 *n*-hexane-acetone mixed solvent: the volume ratio of *n*-hexane to acetone is 1:1.

5.3 Instruments and equipment

5.3.1 Gas chromatograph-mass spectrometer (GC/MS): equipped with electron impact (EI) ion source.

5.3.2 Chromatographic column: 50 % phenylmethylpolysiloxane stationary phase, such as 30 m × 0.25 mm × 0.25 μm or equivalent chromatographic column.

5.3.3 Micro-syringe or autosampler.

5.3.4 Analytical balance: accuracy of 0.01 mg.

5.3.5 Extractor: made of hard glass, tubular, with ground mouth and bottle stopper, 25 mL.

5.3.6 Ultrasonic generator: working frequency of 40 kHz.

5.3.7 Polytetrafluoroethylene membrane filter head: 0.45 μm.

5.3.8 Centrifuge tube with ground mouth and stopper: 10 mL.

5.3.9 Centrifuge: 4000 r/min.

5.4 Gas chromatography-mass spectrometry analysis conditions

5.5.2 Phthalate single standard intermediate solution

Pipette 0.1 mL of each phthalate single standard stock solution into a 10 mL volumetric flask, dilute to the mark with *n*-hexane or other suitable solvents to prepare a single standard intermediate solution. The mass concentration of diisononyl phthalate (DINP) and diisodecyl phthalate (DIDP) solutions is 50 mg/L, and the mass concentration of the remaining substances is 10 mg/L.

NOTE: The standard intermediate solution is sealed and stored in a refrigerator at 0 ℃ ~ 4 ℃, and the validity period is 6 months.

5.5.3 Standard working solution

Use the phthalate single standard intermediate solution to prepare a single standard working solution or a mixed standard working solution. It can prepare the appropriate concentration according to the content of the target substance in the sample.

NOTE: The standard working solution is sealed and stored in a refrigerator at 0 ℃ ~ 4 ℃, and the validity period is 1 month.

5.5.4 Sample solution

Weigh 1.0 g of sample (accurate to 0.0001 g), place in an extractor, accurately add 10.0 mL of *n*-hexane-acetone mixed solution or other suitable extractant, shake well, and extract in an ultrasonic generator for 20 minutes and ensure complete extraction. Use a 0.45 μm polytetrafluoroethylene membrane filter to inject and filter the extract into a small sample bottle for chromatographic analysis.

If the sample is water-soluble, when necessary, add 2 mL of water to the sample first, shake evenly, and then add 10.0 mL of *n*-hexane-acetone mixed solution or other suitable extractant; if the sample is turbid after ultrasonic extraction, use a centrifugal device to centrifuge until the layers are separated and then take the upper clear liquid for analysis.

Prepare a blank sample at the same time.

5.6 Determination

5.6.1 Qualitative analysis

By comparing the retention time and relative abundance of characteristic ions of the target compound in the sample and the standard working solution, it requires the signal-to-noise ratio (*S*/*N*) of the chromatographic peak of the tested specific phthalate to be greater than 3, the retention time of the specific phthalate in the sample is consistent with the retention time of the target compound in the standard solution, and the corresponding characteristic ion abundance ratio of the specific phthalate in the tested

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