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Determination of short chain chlorinated paraffins in textiles dyeing and finishing auxiliaries

纺织染整助剂产品中短链氯化石蜡的测定

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

1 Scope

This Standard specifies the principle, reagents and materials, instruments and equipment, determination steps, blank test, test data processing as well as lower limit of determination, recovery rate, precision and test report for determination of short chain chlorinated paraffins in textiles dyeing and finishing auxiliaries.

This Standard is applicable to the determination of content of short chain chlorinated paraffins C_{10}~C_{13}) in textiles dyeing and finishing auxiliaries.

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

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

3 Principle

Use n-hexane to shake and extract the sample. Purify the extract and set constant volume. Through palladium catalytic dichlorination, short chain chlorinated paraffins become C_{10}~C_{13} normal alkanes. Use a gas chromatograph (GC-FID) equipped with a flame ionization detector to test. Use external standard method of standard short chain chlorinated paraffins with a

flasks. Use n-hexane (4.3) to dilute to the scale then obtain 100μg/mL mixed standard stock solution.

NOTE: Store the standard stock solution at 0°C~4°C. It shall be valid for 12 months.

4.15 Mixed standard working solution: Respectively draw appropriate amount of n-alkane mixed standard stock solution (4.14). Use n-hexane (4.3) to dilute and prepare 0.5μg/mL, 1.0μg/mL, 2.0μg/mL, 5.0μg/mL, 10.0μg/mL, 20.0μg/mL mixed standard working solutions.

NOTE: Store the mixed standard working solution at 0°C~4°C. It shall be valid for 3 months.

4.16 Glass beads with palladium catalyst: Weigh 0.04g of palladium chloride (4.6) and add 5mL of acetic acid solution (4.13). Under slow stirring, use boiling water bath to fully dissolve palladium chloride. Then transfer to an evaporating dish containing 9.5g of glass beads (4.20) (use a small amount of water to wash and transfer to an evaporating dish). Place the evaporating dish on the water bath. In the boiling water bath, heat and stir to evaporate the water. Add water to immerse the glass beads. Use ammonia (4.1) to adjust the pH to 8.8~9.1. Use the boiling water bath to evaporate the water again. Transfer the glass beads with palladium catalyst on the surface to a 100mL sand core funnel. Use 25mL cyclopentane (4.5) to rinse the glass beads. Dry the glass beads for later use.

4.17 Deactivated splitless liner: Match with chromatograph inlet.

4.18 Reaction liner: In the deactivated splitless liner (4.17), load 5mm silanized glass wool (4.22), 2mm calcium carbonate (4.7), 20mm glass beads (4.16) with palladium catalyst and 5mm silanized glass wool (4.22) from bottom to top. The filling height of the liner shall be such that the injection needle does not touch the palladium catalyst layer during injection. Otherwise, the filling height shall be adjusted. The filled liner needs to be aged for 1h at the inlet of the gas chromatograph at 300°C before use. Use standard working solution of short chain chlorinated paraffins (4.11) to determine the catalytic efficiency r of the reaction liner. Guarantee r≥80%.

4.19 pH test paper: precision pH test paper, measuring range: 8.0~9.7.

4.20 Glass beads: diameter is 180μm~250μm.

4.21 Solid phase extraction cartridge: Flori silica, 1g/6mL. It shall be rinsed and activated with 5mL of n-hexane (4.3) before use.

4.22 Silanized glass wool: the highest temperature resistance is 500°C.

5 Instruments and equipment

5.1 Gas chromatograph: equipped with hydrogen flame ionization detector (FID).

5.2 Oscillator: oscillation frequency is (260±30) r/min.

5.3 Vortex mixer.

5.4 Centrifuge: speed is controlled stable at 3000r/min.

5.5 Water bath.

5.6 Nitrogen blowing instrument.

5.7 Centrifuge tube with stopper: 50mL.

5.8 Glass centrifuge tube with stopper: 10mL.

5.9 Analytical balances: resolutions are 0.0001g and 0.01g.

5.10 Sand core funnel: 100mL.

6 Determination steps

6.1 Extraction

Weigh 1.0g of (to the nearest of 0.01g) sample into a 50mL centrifuge tube with stopper (5.7). Add 20mL of n-hexane (4.3). Use an oscillator to oscillate and extract for 30min. Centrifuge the extract with a centrifuge at 3000r/min for 5min. Place still. Take 10mL of the upper n-hexane solution. Transfer to the glass centrifuge tube with stopper (5.8). Concentrate and dilute to 2mL by nitrogen blowing at 40°C. The concentrate is to be purified.

6.2 Purification

Add 5mL of concentrated sulfuric acid (4.8) to the concentrated solution after extraction. Vortex for 5min at room temperature. Sulfonate to transparent solution. Centrifuge the sulfonated solution at 3000r/min for 5min. Discard the concentrated sulfuric acid layer. Add another 5mL of water. Vortex for 5min at room temperature. Take the n-hexane layer for testing.

NOTE: Perform further purification if necessary. Take 1mL of sulfonated upper layer solution. Transfer to the pre-activated solid phase extraction cartridge (4.21). Use 2mL of n-hexane (4.3) to rinse first. Discard the eluent. Then use 5mL of elution solution (4.9) to elute. Collect the eluate. Concentrate to dryness by blowing nitrogen. Use 1mL of n-hexane

solution, in micrograms per milliliter (μg/mL);

c_{oi} - Numerical value of n-alkane concentration converted from blank sample solution, in micrograms per milliliter (μg/mL);

V - Value of the final constant volume, in milliliters (mL);

m - Value of sample mass, in grams (g);

f - Dilution factor;

0.461 - Conversion factor of short chain chlorinated paraffins with chlorine content z = 55.5% (mass fraction).

The calculation result is expressed as the arithmetic mean of the two parallel determination results. Round off according to the comparison method of rounding value in 4.3.3 of GB/T 8170-2008, expressing to one decimal place. When it is lower than the lower limit of determination (9.1), the test result shall be "not detected".

9 Lower limit of determination, recovery rate, precision

9.1 Lower limit of determination

The lower limit of determination of this method is 20mg/kg.

9.2 Recovery rate

The recovery rate of this method for short chain chlorinated paraffins is greater than 80% [calculated as short chain chlorinated paraffins with an average chlorine content of 55.5% (mass fraction)].

9.3 Precision

In the same laboratory, the same operator uses the same equipment, according to the same test method, in a short period of time, the absolute difference between the two independent test results obtained by independent tests on the same object under test is not more than 20% of the arithmetic mean of the two measured values. It is assumed that the arithmetic mean value of these two measured values does not exceed 20% and this situation shall not exceed 5%.

10 Test report

The test report shall at least give the following information:

a) Sample source and description;

Annex B

(normative)

Qualitative confirmation analysis method

B.1 General

This appendix is used for further qualitative confirmation analysis on the test results of palladium-catalyzed hydrogen flame gas chromatography.

B.2 Method principle

Because short chain chlorinated paraffins contain a lot of halogen elements, have very high response on the electron capture detector, and it can eliminate the interference of other compounds that do not contain halogen elements. But the negative chemical source GC/MS instrument can select the characteristic ion scanning of short chain chlorinated paraffins, which has high selectivity.

Use gas chromatograph equipped with electron capture detector or GC/MS with negative chemical source. Perform qualitative confirmation based on retention time and shape of co-eluting peak.

B.3 Instruments and equipment

Gas chromatograph equipped with electron capture detector or GC/MS with negative chemical source.

B.4 Chromatography / mass spectrometry conditions

B.4.1 Analysis conditions of gas chromatography-electron capture detector

Select the best analysis conditions according to different equipment. The reference conditions for analysis of gas chromatography-electron capture detector are as follows:

a) Chromatographic column: DB-5, (30m×0.25mm×0.25μm), or equivalent;

b) Chromatographic column temperature: $60\ ℃ \xrightarrow{20\ ℃/\min} 300\ ℃ (12\ \min)$;

c) Inlet temperature: 280°C (using conventional split liner);

d) Detector temperature: 300°C;

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