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**Determination of organic halogen content
in textile dyeing and finishing auxiliaries**

纺织染整助剂产品中有机卤素含量的测定

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Determination of organic halogen content in textile dyeing and finishing auxiliaries

WARNING -- Personnel using this document shall have practical experience in regular laboratory work. This document does not identify all possible security issues. Users are responsible for taking appropriate safety and health measures and ensuring compliance with the conditions specified in relevant national regulations.

1 Scope

This document describes the method for the determination of organic halogen content in textile dyeing and finishing auxiliaries.

This document applies to the determination of organic halogen content in various types of textile dyeing and finishing auxiliaries, including organic fluorine, organic chlorine, and organic bromine.

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

3 Terms and definitions

For the purpose of this document, the following term and definition apply.

3.1

organic halogen

Halogens combined with organic compounds.

NOTE 1: The organic halogen compounds in this document include organic compounds of fluorine (F), chlorine (Cl), and bromine (Br), except iodine (I).

NOTE 2: Under the test conditions of this document, some inorganic halogen compounds will also be determined as organic halogens if they are not removed from the test.

4 Principle

Disperse or dissolve the sample in a nitric acid aqueous solution, use activated carbon to adsorb the organic halide in the solution, and put the activated carbon adsorbed with the organic halide into a high-temperature furnace for combustion, cracking, and gasification. Its product, hydrogen halide (hydride of fluorine, chlorine, bromine), is absorbed by alkaline aqueous solution, and then the halogen ions are separated and determined by ion chromatography and quantified by the external standard method.

5 Reagents or materials

Unless otherwise specified, only use reagents confirmed to be of analytical grade and first-grade water specified in GB/T 6682. It is recommended that the containers used be cleaned with nitric acid solution (5.8) before use, and then rinsed with water.

5.1 Activated carbon: granular (0.5 mm ~ 2.5 mm). See Annex A for purification preparation method.

5.2 Oxygen: purity greater than or equal to 99.995 % (volume fraction).

5.3 Argon gas: purity greater than or equal to 99.99 % (volume fraction).

5.4 Nitric acid.

5.5 Potassium hydroxide, guaranteed reagent.

5.6 Anhydrous sodium carbonate, guaranteed reagent.

5.7 Sodium bicarbonate, guaranteed reagent.

5.8 Nitric acid solution, 1 mol/L: PIPETTE 67 mL of nitric acid (5.4), ADD water to make the volume to 1000 mL.

5.9 Sodium nitrate solution, 0.85 g/L: WEIGH 17 g of sodium nitrate and DISSOLVE it in a small amount of water, ADD 1.4 mL of nitric acid (5.4), TRANSFER it to a 1000 mL volumetric flask, and DILUTE to the mark with water. Then use a pipette to transfer 50 mL of the solution, and then dilute to 1000 mL with water.

5.10 Organic halogen reference solutions, 0.25 mg/mL: WEIGH 184.4 mg of 4-fluorobenzoic acid, 110.3 mg of 4-chlorobenzoic acid, and 62.9 mg of 4-bromobenzoic acid, ADD water to dissolve respectively and DILUTE to 100 mL to prepare reference solutions of organic fluorine, organic chlorine and organic bromine with mass concentrations of 0.25 mg/mL. Dilute as needed when using.

NOTE: Stored in an airtight plastic container at 4 °C, the validity period is 12 months.

5.11 Potassium hydroxide solution, 30 mmol/L: WEIGH 1.68 g of potassium hydroxide (5.5), DISSOLVE it in water and DILUTE to 1000 mL.

5.12 Mixed solution of 3.2 mmol/L sodium carbonate and 1.0 mmol/L sodium bicarbonate: WEIGH 0.3392 g of anhydrous sodium carbonate (5.6) and 0.0840 g of sodium bicarbonate (5.7), DISSOLVE with water and DILUTE to 1000 mL.

5.13 Halogen ion mixed standard stock solution, 1 mg/mL: BAKE the reference reagents of sodium fluoride, sodium chloride, and sodium bromide at 105 °C for 120 min (or burn at 500 °C for 30 min), COOL and PLACE in a desiccator. WEIGH 2.2105 g of sodium fluoride, 1.6485 g of sodium chloride, and 1.2877g of sodium bromide after drying, DISSOLVE them in water and DILUTE to 1000 mL. The mass concentrations of F⁻, Cl⁻, and Br⁻ ions in the stock solution are all 1 mg/mL. Or purchase commercially available standard solutions.

NOTE: Stored in an airtight plastic container at 4 °C, the validity period is 12 months.

5.14 Halogen ion mixed standard working solution: DILUTE the halide ion mixed standard stock solution (5.13) with water, and prepare halide ion mixed standard working solution with an appropriate series of concentrations as needed. It is recommended that the mass concentration of F⁻, Cl⁻, and Br⁻ ions be in the range of 0.10 mg/L ~ 2.00 mg/L.

NOTE: Stored in an airtight plastic container at 4 °C, the validity period is 12 months.

6 Instruments and equipment

6.1 Ion chromatograph: equipped with conductivity detector.

6.2 High-temperature combustion device: It consists of a tube furnace, a cracking tube, a quartz boat, and a controller. The schematic diagram is shown in Figure 1, or equivalent. The tube furnace can control the temperature within the range of 500 °C ~ 1000 °C, with an error of less than 10 °C.

Halogen-free gloves shall be worn during sample collection, and samples shall be sealed and stored in polyethylene bags or bottles. If the sample analysis cannot be completed within 7 days after sampling, it shall be stored frozen to reduce biodegradation and reduce the loss of volatile halogen compounds.

Take at least 0.5 g of a representative sample, and weigh no less than 2 homogeneous samples of 10 mg ~ 50 mg (accurate to 0.1 mg).

7.2 Activated carbon adsorption

7.2.1 PUT the weighed sample into a 100 mL Erlenmeyer flask (6.3) with a ground stopper, ADD 50 mL of sodium nitrate solution (5.9), and ADJUST the pH value to less than 2 with nitric acid solution (5.8).

7.2.2 ADD 50 mg of activated carbon (5.1) to the Erlenmeyer flask, COVER it with a ground stopper, PLACE it in the oscillator (6.4) to oscillate and absorb at room temperature for 12 hours. After adsorption, TRANSFER the solution and activated carbon to the filtration device (6.5) for filtration, and then WASH the activated carbon particles several times with 25 mL of sodium nitrate solution (5.9). Then PUT the activated carbon particles into the quartz boat and proceed with combustion and cracking according to step 7.3.

7.3 Combustion and cracking

7.3.1 WARM UP the tube furnace in advance, ADJUST the oxygen flow rate to more than 160 mL/min, and VENTILATE for more than 10 minutes to stabilize the instrument.

7.3.2 CONNECT the outlet end of the cracking tube to the absorption tube (6.6) containing 10 mL of potassium hydroxide solution (5.11).

7.3.3 MOVE the quartz boat filled with activated carbon particles to the dry area of the combustion tube, LET IT STAND for more than 5 minutes. There shall be no water condensation in the low temperature area of the combustion tube.

7.3.4 PUSH the quartz boat into the high-temperature area of the combustion tube (set to 950 °C) for full combustion and cracking. The duration shall not be less than 3 minutes.

7.3.5 MOVE the quartz boat to the low temperature area and continue to blow oxygen for cooling for more than 2 minutes. TAKE OUT the absorption tube, FILTER the absorption solution through the needle filter head (6.9), for determination.

7.4 Analytical methods

7.4.1 Chromatographic conditions

The best analysis conditions can be selected according to different instruments and equipment. The reference conditions for chromatographic analysis are as follows.

a) Chromatographic conditions one:

- Chromatographic column: hydroxide selectivity, anion exchange column, 250 mm × 4 mm (inner diameter);
- Column temperature: 30 °C;
- Eluent: potassium hydroxide solution (5.11);
- Flow rate: 1.0 mL/min;
- Sample injection volume: 25 μL.

b) Chromatographic conditions two:

- Chromatographic column: carbonate selectivity, anion exchange column, 150 mm × 4 mm (inner diameter);
- Column temperature: 30 °C;
- Eluent: a mixed solution of 3.2 mmol/L sodium carbonate and 1.0 mmol/L sodium bicarbonate (5.12);
- Flow rate: 0.7 mL/min;
- Sample injection volume: 20 μL.

7.4.2 Qualitative analysis

Analyze the halide ion mixed standard working solution (5.14) and absorption solution (7.3.5) in sequence according to the analysis conditions of 7.4.1, and conduct qualitative analysis based on the retention time of F⁻, Cl⁻ or Br⁻ ions. The halide ion separation chromatogram of the standard working solution is shown in Figure B.1 and Figure B.2 in Annex B.

7.4.3 Quantitative analysis

Plot a standard working curve with the mass concentration of F⁻, Cl⁻ or Br⁻ ions in the halide ion mixed standard working solution (5.14) as the abscissa and the corresponding peak area as the ordinate. According to the chromatographic peak area of F⁻, Cl⁻ or Br⁻ ions in the absorption solution (7.3.5), use the external standard method for quantification. The response value of the target substance in the sample solution shall be within the linear range of testing of the instrument. If the content exceeds the range of the standard working curve, the sample solution shall be diluted to an appropriate concentration before analysis.

The lower limit of determination of organic fluorine, organic chlorine or organic bromine in this document is 25.0 mg/kg.

9.2 Recovery rate

The standard-adding recovery rates of organic fluorine, organic chlorine or organic bromine in this document are all 85 % ~ 110 %.

9.3 Precision

The absolute difference between the two independent test results obtained in the same laboratory, by the same operator using the same equipment, according to the same test method, by testing the same tested object independently of each other in a short period of time, is not greater than 10 % of the arithmetic mean of the two measured values, provided that the case, where it is greater than 10 % of the arithmetic mean of the two measured values, does not exceed 5 %.

10 Test report

The test report should at least provide the following content:

- a) sample source and description;
- b) serial number of this document;
- c) test conditions;
- d) test results;
- e) any deviation from the details of this document;
- f) test date.

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