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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 42908-2023

**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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Foreword

This document was issued on 6 August 2023 by the State Administration for Market Regulation; Standardization Administration of the PRC and takes effect on 1 March 2024.

It is a GB/T standard: recommended rather than compulsory, but it is the text a Chinese reviewer applies when assessing a submission.

1 Scope

GB/T 42908-2023 gives the determination of organic halogen content in textile dyeing and finishing auxiliaries. Organic halogens reach a fabric through the auxiliaries rather than the dyes - the fluorinated water repellents, the chlorinated carriers and the brominated flame retardants that are added at the finishing stage - and they are the substances most restricted by international buyers, so a mill needs to know what is in the drum before it goes into the bath. The standard describes the principle, the reagents and materials, the instruments and equipment, the test procedure, the processing of the data, the lower limit of determination, the recovery and precision, and the test report, with informative annexes on the purification of the activated carbon and on the halide ion separation chromatogram. It covers organic fluorine, chlorine, bromine and iodine and took effect on 1 March 2024.

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).