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Textiles – Determination of Adsorbable Organic Halogens

纺织品 可吸附有机卤素的测定

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Table of Contents

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Warning: Personnel using this Document shall have practical working experience in formal laboratory. This Document does not address all possible security issues. It is the user's responsibility to take appropriate safety and health measures and ensure compliance with the conditions stipulated in the relevant national laws and regulations.

1 Scope

This Document describes a test method for the determination of adsorbable organic halogens in textiles by ion chromatography.

This Document applies to all types of textile products.

2 Normative References

The provisions in following documents become the essential provisions of this Document through reference in 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 Document.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

3 Terms and Definitions

For the purposes of this Document, the following terms and definitions apply.

3.1 Adsorbable organic halogens

Under specified conditions, the total amount of halogens (including fluorine, chlorine, bromine and iodine) combined with organic compounds that can be adsorbed by activated carbon.

4 Principle

The specimen is ultrasonically extracted and filtered. The organic halides in the filtrate are adsorbed by activated carbon; and then the inorganic halides are separated by washing with acidic sodium nitrate solution. The activated carbon adsorbed by the organic halides is burned, cracked and gasified in an oxygen flow; the product of hydrogen halide enters the absorption liquid with the gas; and the halogen ion is analyzed and determined by ion chromatograph (IC); and the external standard method is used for quantification.

5 Reagents and Materials

Unless otherwise stated, only guaranteed reagents are used. The used reagents and materials shall not contain the halogens to be tested.

5.1 Water

It shall meet the requirements of Class-I water specified in GB/T 6682.

5.2 Halide ion standard stock solution

5.2.1 Fluoride ion standard stock solution

Prepare a fluoride ion standard stock solution with a mass concentration of 1000mg/L: Take 2.2105g of sodium fluoride dried at 100°C ~ 105°C to constant weight; dissolve it in water; make constant volume to 1000mL; store in polypropylene or high-density polyethylene bottles; then place them in a refrigerator at 0°C ~ 4°C.

5.2.2 Chloride ion standard stock solution

Prepare a chloride ion standard stock solution with a mass concentration of 1000 mg/L: Take 1.6480 g of sodium chloride burned at 500 °C ~ 600 °C to constant weight; dissolve it in water; make constant volume to 1000 mL; store in a brown bottle; and place it in a refrigerator at 0°C~4°C.

5.2.3 Bromide ion standard stock solution

Prepare a bromide ion standard stock solution with a mass concentration of 1000 mg/L: Take 1.4880 g of potassium bromide that has been dried at 100 °C ~ 105 °C to constant weight; dissolve it in water; make constant volume to 1000 mL; store in a brown bottle; and place it in a refrigerator at 0°C~4°C.

5.2.4 Iodide ion standard stock solution

Prepare an iodide ion standard stock solution with a mass concentration of 1000 mg/L: Take 1.3073 g of dried potassium iodide; dissolve in water; make constant volume to 1000 mL; store in a brown bottle; and place it in a refrigerator at 0°C ~ 4°C.

NOTE 1: The storage period of the standard stock solution is 6 months at 0°C~ 4°C.

NOTE 2: Commercially available certified standard solutions can also be used in this experiment.

5.3 Halide ion mixed standard working solution

6 Apparatus

6.1 Ion chromatograph (IC): Equipped with a digital conductivity detector.

6.2 Combustion absorbing device: It is mainly composed of tube furnace, combustion tube, quartz boat, flow controller, temperature controller and porous absorption bottle. The temperature adjustment range is from room temperature to 1000°C; and the temperature control accuracy is ±1%; see Appendix A for its structure. Or there are combustion absorbing devices or equipment with similar functions.

6.3 Ultrasonic cleaner: Frequency 40kHz, power ≥ 200W.

6.4 Mechanical oscillator: Reciprocating 150 times/min~250 times/min; amplitude 6cm~10cm; its stirring force shall be adjustable; while keeping stirring the specimen, it shall ensure that the specimen shall not overflow.

6.5 Filtering device: Including vacuum suction filtration, filter cup, sand core filter head, and liquid collection bottle.

6.6 Conical flask: 250mL, with a standard ground glass stopper.

6.7 Analytical balance: Sensitivity of 0.01g and 0.1mg.

6.8 Porous absorption bottle: 50mL.

6.9 Sand core funnel: Model of G2.

7 Analysis Procedures

7.1 Preparation of specimen

Take a representative sample; cut it into pieces below 5mm×5mm; mix well; put it in a plastic bag or a glass bottle and store it.

NOTE: Wear protective gloves during sample preparation to prevent sample contamination; the used plastic bags and gloves must not contain the halogen to be tested.

7.2 Extraction

7.2.1 Take two portions of specimens (for parallel tests), 2g for each portion, accurate to 0.01g. Place them in conical flasks (6.6); add 100mL of water (5.1); cap the bottle tightly and vigorously shake the conical flask so that all the specimens are immersed in the solution. Put the conical flask into the ultrasonic cleaner (6.3); and conduct ultrasonic extraction at (25±5) °C for 1h.

7.2.2 Filter by a sand core funnel (6.9); wash the specimen and conical flask (6.6) with 25mL of water (5.1); and collect all the filtrate in another clean conical flask (6.6).

7.2.3 Use nitric acid (5.4) to adjust the pH value of the above-mentioned filtrate to below 3; add 50 mg of activated carbon (5.8); cap the bottle tightly and place it on a mechanical oscillator (6.4); and shake at room temperature at a speed of 150 times/min for 1h.

7.2.4 Use polycarbonate filter membrane (5.9) to filter by filtering device (6.5); rinse activated carbon (5.8), inner walls of conical flask (6.6) and filter cup (6.5) by 25mL of acidic sodium nitrate working solution (5.6). And then wash the activated carbon (5.8) with a small amount of water (5.1); vacuum filter it to nearly dry; remove the treated activated carbon (5.8) together with the filter membrane; and use them for combustion.

7.3 Combustion and absorption

7.3.1 Pre-set the tube furnace temperature of the combustion absorbing device (6.2) and keep it at (950±10) °C.

7.3.2 Add 10mL of eluent (5.7) to the two porous absorption bottles (6.8) respectively for secondary absorption of gas products.

7.3.3 Adjust the oxygen (5.11), pressure and flow meter so that the oxygen flow rate of the inner sleeve of the combustion tube is 120mL/min~150mL/min; the oxygen flow rate of the outer tube is 40mL/min~60mL/min; and the bubbles in the absorption bottle can be blown out evenly.

7.3.4 Put the activated carbon treated in 7.2.4 together with the filter membrane into the quartz boat and push it into the low temperature zone (at the furnace mouth) of the tube furnace; stay for 2 min; and then slowly push the quartz boat into the high temperature zone of the tube furnace (middle position); pull it back after 5 min; and continue blowing oxygen for 5 min.

7.3.5 Remove the secondary porous absorption bottle (6.8); quantitatively transfer the absorption liquid to a 25mL volumetric flask; continue to wash the porous absorption bottle (6.8) with a little eluent and make constant volume to the mark; and pass through the inorganic filter membrane (5.10); and the filtrate is used for instrumental analysis.

7.4 Instrumental analysis

Analyze the specimen solution (7.3.5) and the mixed standard working solution (5.3) of fluorine, chlorine, bromine and iodide ions. See Appendix B for instrument analysis conditions. Perform qualitative analysis by comparing the retention time of the specimen solution (7.3.5) with the halide ion mixed standard working solution (5.3). Take the chromatographic peak area of the standard substance as the ordinate, and the mass concentration of the standard working solution as the abscissa to draw the standard working curve; and the external standard method is used for quantitative analysis.

The typical ion chromatograms of fluorine, chlorine, bromine and iodine can be seen in Figure B.1.

Appendix B

(Informative)

Ion Chromatography Analysis of Fluorine, Chlorine, Bromine and Iodine

B.1 Ion chromatography analysis conditions

Different instruments are used; and the optimal analysis conditions may also be different; so, it is impossible to give general parameters for ion chromatography analysis. The set parameters shall ensure that the measured component and other components can be effectively separated during chromatographic determination. The parameters given below have been proven feasible:

a) Chromatographic column: High-capacity anion exchange column; 250mm×4mm (inner diameter) and guard column; 50mm×4mm (inner diameter), or equivalent;

b) Eluent: 3.2mmol/L Na_2CO_3 + 1.0mmol/L $NaHCO_3$;

c) Flow rate: 0.7mL/min;

d) Column temperature: 35°C;

e) Detector: Digital conductivity detector;

f) Injection volume: 20μL.

B.2 Ion chromatograms

The typical ion chromatograms of fluorine, chlorine, bromine and iodine are shown in Figure B.1.

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