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

SN/T 5236-2020

**Import and export textiles Determination of harmful dyes Liquid
chromatography-high resolution mass spectrometry**

进出口纺织品 有害染料的测定 液相色谱-高分辨质谱法

(English Translation)

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

1 Scope

This standard specifies the use of liquid chromatography-high resolution mass spectrometry (LC-HRMS) for the simultaneous determination of 46 hazardous dyes in textile products.

method.

This standard applies to the determination of 46 harmful dyes in textile products processed by printing and dyeing.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version applies to this article. For undated reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 6682 Analytical laboratory water specifications and test methods

3 Principle

The sample was heated and extracted with pyridine-aqueous solution as the extraction solvent, the supernatant was diluted, and then liquid chromatography-high resolution mass spectrometry (LC-HRMS) determination, external standard method quantification.

4 Reagents and materials

Unless otherwise stated, all reagents used are of analytical grade. The water is the first grade water that meets the requirements of GB/T 6682.

4.1

Acetonitrile. chromatographically pure.

4.2

Methanol. chromatographically pure.

4.3

Pyridine. excellent grade pure.

4.4

Ammonium acetate. chromatographically pure.

4.5

Formic acid. chromatographically pure.

4.6

Aqueous solution of ammonium acetate (0.385 g/L). accurately weigh 0.385 g of ammonium acetate (4.4) and dissolve in water, and transfer to a 1000 mL volumetric flask.

Constant volume with water.

4.7

Ammonium acetate aqueous solution (0.385 g/L, containing 0.01% formic acid). accurately weigh 0.385 g of ammonium acetate (4.4) and dissolve in water, and add 100 μ L Formic acid (4.5), transfer to a 1000 mL volumetric flask, and dilute to volume with water.

4.8

Pyridine-aqueous solution (11, volume ratio). equal volume of pyridine (4.3) is mutually soluble in water and stored in a brown glass bottle.

4.9

Methanol-aqueous solution (11, volume ratio). equal volume of methanol (4.2) and water are mutually soluble.

4.10 46 kinds of dye standards. see Appendix A.

4.11

Standard stock solution. accurately weigh an appropriate amount of dye standard and dissolve it with the solvent listed in Appendix A to make an effective concentration of 100 mg/L Standard stock solution. The validity period of the standard stock solution is 12 months when stored in a refrigerator at -18 °C and protected from light.

4.12

Mixed standard working solution. accurately pipette an appropriate amount of standard stock solution and use methanol-aqueous solution (4.9) to prepare a concentration of 1 mg/L the mixed standard working solution of, or the standard working solution of other concentration can be prepared as needed. Stored in the refrigerator at -18 °C and protected from light, the validity period of the standard working solution is 1 month.

5 Apparatus and equipment

5.1

Liquid Chromatography-High Resolution Mass Spectrometer (LC-HRMS). Distributed Spray Ion Source (ESI).

5.2

Heating source. equipment that can generate a heat source of $(100 \pm 2)^\circ\text{C}$, such as a water bath placed on an electric hot plate and heated to boiling.

5.3

Glass sample bottle. 40 mL with screw cap (with PTFE gasket).

5.4

Electronic balance. Sensitivity 0.01 g and 0.1 mg.

5.5

Microporous membrane. 0.22 μm , polytetrafluoroethylene.

6.1 Sample preparation and extraction

Take a representative sample, cut into pieces of about 5 mm x 5 mm, and mix well. Accurately weigh 1 g (accurate to 0.01 g) sample at 40 Add 20 mL of pyridine-water mixed solution (4.8) to the mL glass bottle (5.3), and extract at $(100 \pm 2) ^\circ\text{C}$ for 35 min. After the extraction is complete, Cool to room temperature, take the supernatant and dilute 10 times with methanol-water mixed solution (4.9), pass through a 0.22 μm microporous membrane, and wait for the instrument to inject the sample.

6.2.1.1 Liquid chromatography conditions

6.2.1.1.1

Chromatographic column. CAPCELL PAK UG120 C181), 3 μm , 2.0 mm x 100 mm, or equivalent;

6.2.1.1.2

Mobile phase. In positive ion mode. A is 5 mmol/L ammonium acetate aqueous solution (containing 0.01% formic acid) (4.7), B it is acetonitrile (4.1); in negative ion mode. A is 5 mmol/L ammonium acetate aqueous solution (4.6), and B is acetonitrile (4.1). Positive and negative ion mode the gradient elution procedure is the same, see Table 1;

6.2.1.1.3

Flow rate. 0.25 mL/min;

6.2.1.1.4

Injection volume. 5 μL ;

6.2.1.1.5

Column temperature. 30 $^\circ\text{C}$.

6.2.1.2 High resolution mass spectrometry conditions

6.2.1.2.1

Ion source. electrospray ion source;

1) This information is given for the convenience of users of this standard, and does not mean approval of the product. If other equivalent products have the same effect, these equivalent products can then be used.

6.2.1.2.2

Scan mode. positive and negative ion mode, collect separately;