

ICS 59.080.01
CCS W 04

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

SN/T 5237-2020

**Import and export textiles. Determination of extractable heavy
metals. Inductively coupled plasma mass spectrometry**

进出口纺织品 可萃取重金属的测定 电感耦合等离子体质谱法

(English Translation)

Issued on: August 27, 2020

Implemented on: March 1, 2021

Issued by: General Administration of Customs

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Foreword

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

This standard was proposed and managed by the General Administration of Customs of the People's Republic of China.

1 Scope

This standard specifies the use of inductively coupled plasma mass spectrometer (ICP-MS) to extract heavy metals such as lead (Pb) and cadmium in textiles.

(Cd), Chromium (Cr), Arsenic (As), Copper (Cu), Antimony (Sb), Nickel (Ni), Cobalt (Co), Mercury (Hg) 9 simultaneous determination method.

This standard applies to the determination of extractable heavy metals in various textile materials and their products.

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 Pieces. For undated

reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 602 Preparation of standard solutions for impurity determination of chemical reagents
GB/T 3922 Textile color fastness test Color fastness to perspiration GB/T 6682 Analytical laboratory water specifications and experimental methods

3 Principle

The sample was extracted with artificial acid sweat, filtered, and measured with an inductively coupled plasma mass spectrometer. Qualitative by element specific mass number Analysis, quantitative analysis with internal standard method.

4 Reagents and materials

Unless otherwise specified, all reagents used are pure superior grade, and the experimental water is secondary water that meets the requirements of GB/T 6682.

4.1

Potassium dichromate.

4.2

Hydrochloric acid. The mass concentration is 36%~38%.

4.3

Nitric acid. The mass concentration is 65%~68%.

4.4 2% hydrochloric acid solution (volume concentration).

4.5 1% nitric acid solution (volume concentration).

4.6 5% nitric acid solution (volume concentration).

4.7 20% nitric acid solution (volume concentration).

4.8

Nitric acid solution of potassium dichromate (0.5 g/L). Weigh 0.05 g of potassium dichromate (4.1) and dissolve in 100 mL of 5% nitric acid solution (4.6).

4.9

Acid sweat. Use the secondary water specified in GB/T 6682 to prepare acid sweat according to GB/T 3922, and use it now.

4.10

Lead, cadmium, chromium, arsenic, copper, antimony, nickel, cobalt, mercury standard stock solution (1000 mg/L). prepared according to GB/T 602 standard or Purchase certified

reference materials directly.

4.11

Mixed standard working solution (10 mg/L). accurately pipette 0.5 mL standard stock solution of lead, cadmium, chromium, arsenic, copper, antimony, nickel, and cobalt.

The solution (4.10) is placed in a 50 mL volumetric flask, and the volume is fixed with 5% nitric acid solution (4.6) to obtain a 10 mg/L mixed standard working solution.

Store in the refrigerator away from light, and the validity period is 1 month.

4.12

Mercury standard working solution (10 mg/L). accurately pipette 0.5 mL of mercury standard stock solution (4.10) into a 50 mL volumetric flask, use the nitric acid solution of potassium dichromate (4.8) is fixed to a volume of 10 mg/L mercury standard working solution, and it is stored in a refrigerator at 4 °C and protected from light. The validity period is 2 years.

4.13

Internal standard stock solution (100 mg/L). Prepare according to GB/T 602 standard or directly purchase certified reference materials, the internal standard of each analysis element for standard elements, see Table A.2.

4.14

Mixed internal standard working solution (1 mg/L). respectively take 0.5 mL internal standard stock solution (4.13) in a 50 mL volumetric flask, and use 1% the volume of nitric acid solution (4.5) was fixed to obtain a mixed internal standard working solution of 1 mg/L.

4.15

Argon gas. purity $\geq 99.999\%$.

4.16

Helium. purity $\geq 99.999\%$.

5 Apparatus and equipment

5.1

Inductively coupled plasma mass spectrometer (ICP-MS).

5.2

Analytical balance. Sensitivity is 0.01 g and 0.1 mg.

5.3

Conical flask with stopper. 150 mL.

Note: Before use, soak in 20% nitric acid solution overnight, rinse with water, and finally rinse with water for more than three times and dry.

5.4

Constant temperature water bath oscillator. It can be controlled at a constant temperature $(37\pm 2)^{\circ}\text{C}$, and the oscillation frequency is 60 times/min.

5.5

Water filter membrane. $0.45\ \mu\text{m}$.

6.1 Preparation of extraction sample solution

Take a representative sample, cut it into small pieces below $5\ \text{mm}\times 5\ \text{mm}$, mix well, weigh $4\ \text{g}$ sample, accurate to $0.01\ \text{g}$, and place it in a stopper.

In the triangular flask (5.3), add $80\ \text{mL}$ of acidic sweat (4.9), soak the fiber fully, and place it in a constant temperature water bath shaker (5.4).

After shaking for $(60\pm 5)\ \text{min}$, take it out, let stand and cool to room temperature, filter through membrane (5.5), add $0.1\ \text{mL}$ per $10\ \text{mL}$ of filtrate for mercury measurement Hydrochloric acid (4.2), add $0.05\ \text{mL}$ of nitric acid (4.3) for every $10\ \text{mL}$ of filtrate to test other elements, as the test solution.

6.2 Blank test

Carry out a blank test with the same product.

6.3 Preparation of standard working curve

Take a certain amount of lead, cadmium, chromium, arsenic, copper, antimony, nickel, and cobalt mixed standard working solution (4.11), use 1% nitric acid solution (4.5) Prepare a series of standard working solutions by diluting step by step. Take a certain amount of mercury standard working solution (4.12) and dilute with 2% hydrochloric acid solution (4.4) step by step Prepare a series of standard working solutions (the highest point does not exceed $5\ \mu\text{g/L}$).

6.4 Determination

Refer to Table A.1 to optimize the parameters of the instrument conditions. The mass number and integration time of the analysis elements are shown in Table A.2.

Solution, blank solution and sample solution. If the measurement result exceeds the range of the standard working curve, the test solution needs to be diluted before proceeding Determination.