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

SN/T 5231-2019

**Leather heavy metals (lead, cadmium, mercury, chromium)
screening method energy dispersive X fluorescence
spectrometry**

(English Translation)

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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.

Drafting organizations of this standard. Jiangmen Customs of the People's Republic of China, Huangpu Customs of the People's Republic of China, Changsha of the People's Republic of China Customs, Shanghai Customs of the People's Republic of China, Guangzhou Customs of the People's Republic of China, Fuzhou University.

1 Scope

This standard specifies the test method for heavy metals (lead, cadmium, mercury, chromium) in leather-energy dispersive X-ray fluorescence spectrometry (ie ED-XRF) law).

This standard applies to the quantitative screening of heavy metals (lead, cadmium, mercury, chromium) in leather.

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 8170 Numerical rounding rules and the expression and determination of limit values
QB/T 2706 Leather chemical, physical, mechanical and color fastness test sampling site

3 Principle

Under the set conditions, the energy dispersive X-ray fluorescence spectrometer is used to determine the content of heavy metals (lead, cadmium, mercury, chromium) in the sample.

Analyze the X-ray fluorescence intensity, and calculate the heavy metal content of the sample according to the quantitative relationship between the X-ray fluorescence intensity and the content of the element to be measured.

content.

4 Reagents and materials

Unless otherwise specified, all reagents used in the analysis are of analytical grade.

4.1

Lead nitrate.

4.2

Cadmium nitrate tetrahydrate.

4.3

Mercury nitrate.

4.4

Chromium nitrate.

4.5

Sodium carboxymethyl cellulose.

4.6

Tetrahydrofuran.

4.7

Lead (Pb) element standard stock (10000 mg/kg). Weigh 0.016 g of lead nitrate (4.1), place

it in a ceramic mortar (5.5), and add 0.984 g Sodium carboxymethyl cellulose (4.5), quickly and fully grind uniformly.

4.8

Cadmium (Cd) element standard stock (10000 mg/kg). Weigh 0.027 g of cadmium nitrate tetrahydrate (4.2) in a ceramic mortar (5.5), add 0.953 g of sodium carboxymethyl cellulose (4.5), and grind quickly and thoroughly.

4.9

Mercury (Hg) element standard stock (10000 mg/kg). Weigh 0.016 g of mercury nitrate (4.3), place it in a ceramic mortar (5.5), and add 0.984 g Sodium carboxymethyl cellulose (4.5), quickly and fully grind evenly.

Note: Mercury nitrate is highly toxic, so care should be taken to protect it during storage and preparation.

4.10

Chromium (Cr) element standard stock (10000 mg/kg). Weigh 0.046 g of chromium nitrate (4.4), place it in a ceramic mortar (5.5), and add 0.954 g Sodium carboxymethyl cellulose (4.5), quickly and fully grind evenly.

4.11

Blank leather samples. no target elements such as lead, cadmium, mercury, and chromium.

5 Apparatus and equipment

5.1

X fluorescence spectrum analyzer (EDX type).

5.2

Electronic balance. accuracy of 0.1 mg.

5.3

High-speed pulverizer.

5.4

Tablet press. The pressure is not less than 20 tons, and the die diameter is 10-20 mm.

5.5

Ceramic mortar.

5.6

Blender.

5.7

Three-roll mill.

6.1 Series standard preparation

Weigh a certain amount of blank leather sample (4.11), fully cut it to about 2 mm, and then use a high-speed crusher (5.3) to crush to about 0.1 mm.

Add 10000 mg/kg standard stock, then add a certain amount of tetrahydrofuran (4.6), stir evenly with a mixer (5.6), then use three rolls the grinder (5.7) performs grinding and rolling, and then presses the tablet with the tablet press (5.4) to make a standard with a concentration range of 50 mg/kg ~ 1000 mg/kg Standard substance (at least 5 concentrations).

6.2 Preparation of standard working curve

Select the appropriate measurement conditions and energy range according to the instrument and equipment and the measured heavy metals, enter the standard product information, and sequentially set the series of standards the quasi-object is put into the instrument for testing, and the heavy metal standard working curve is prepared, with heavy metal content as the X axis and heavy metal test intensity as the Y axis. X refer to Appendix A for the test conditions of the fluorescence spectrometer.

6.3 Testing

The sample should be representative and meet the requirements of QB/T 2706. Generally, the sample does not need to be cut and sampled and pre-treated to keep the sample surface free the quality can be directly determined. Place the sample in the test window of the instrument. The test conditions and energy range are consistent with the measurement standard. Three points are randomly selected Test, read the heavy metal concentration in the sample against the standard working curve. If the sample is found to be non-uniform, the test point should be increased.

7 Result calculation

Calculate the content of heavy metals in leather according to formula (1).

Where.

C_i - the content of heavy metal elements in the sample, in milligrams per kilogram (mg/kg);

I_i - Test intensity of heavy metal elements, in units per microampere per second (cps/ μ A);

b - Intercept of the standard working curve of heavy metal elements, in units per microampere per second (cps/ μ A);