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GB/T 22807-2019

Replacing GB/T 22807-2008

**Leather and fur - Chemical tests - Determination
of chromium (VI) content: Colorimetric method**

皮革和毛皮 化学试验 六价铬含量的测定：分光光度法

[ISO 17075-1:2017, Leather - Chemical determination of chromium (VI)
content in leather - Part 1: Colorimetric method, MOD]

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Leather and fur - Chemical tests - Determination of chromium (VI) content: Colorimetric method

1 Scope

This Standard specifies the colorimetric method for the determination of chromium content in leather and fur.

This Standard applies to the determination of chromium content in various leather, fur and their products.

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 6682-2008, ISO 3696:1987, MOD)

GB/T 38402 Leather and fur - Chemical tests - Determination of chromium (VI) content: Chromatographic method (GB/T 38402-2019, ISO 17075-2:2017, MOD)

QB/T 1267 Fur - Chemical, physical and mechanical and fastness tests - Sampling location (QB/T 1267-2012, ISO 2418:2002, MOD)

QB/T 1272 Fur - Preparation of chemical test samples (QB/T 1272-2012, ISO 4044:2008, MOD)

QB/T 1273 Fur - Chemical tests - Determination of volatile matter (QB/T 1273-2012, ISO 4684:2005, MOD)

QB/T 2706 Leather - Chemical, physical and mechanical and fastness tests - Sampling location (QB/T 2706-2005, ISO 2418:2002, MOD)

QB/T 2716 Leather - Preparation of chemical test samples (QB/T 2716-2018, ISO 4044:2008, MOD)

QB/T 2717 Leather - Chemical tests - Determination of volatile matter (QB/T

6 Sampling and preparation of samples

6.1 Sampling

For leather, it is carried out according to the provisions of QB/T 2706.

For fur, it is carried out according to the provisions of QB/T 1267.

If the sampling is not possible according to the requirements of QB/T 2706 or QB/T 1267 (such as shoe uppers, leather on leather clothing), the sampling process shall be indicated in the report.

6.2 Preparation of samples

Leather: according to the provisions of QB/T 2716.

Fur: according to the provisions of QB/T 1272. It shall avoid damaging the fur during the sample preparation process and keep the fur intact.

REMOVE the glue and attachments on the sample as cleanly as possible, MIX the sample evenly, and PUT it into a clean sample bag for test.

7 Test procedure

7.1 Preparation of analytical solution

WEIGH (2.0 ± 0.1) g of cut sample, to the nearest 0.001 g.

PIPETTE 100 mL of degassed phosphate buffer (4.2) into a 250 mL conical flask (5.2); INSERT an aeration tube (5.3) (the aeration tube must not touch the liquid surface); PASS argon or nitrogen into the conical flask (4.8) at a flow rate of (50 ± 10) mL/min. After 5 min, REMOVE the aeration tube; ADD the sample and COVER with a ground stopper; PLACE it in an oscillator (5.1) at room temperature ($18\text{ }^{\circ}\text{C} \sim 26\text{ }^{\circ}\text{C}$) for water bath extraction for (180 ± 5) min, and the oscillation frequency is (100 ± 10) times/min.

NOTE 1: Take care to avoid the sample sticking to the flask wall above the liquid surface during the oscillation.

NOTE 2: The extraction conditions have a direct impact on the test results of this method. The results obtained by using different extraction conditions (such as extraction solvent, pH value, extraction time, etc.) are not comparable to the results obtained by this method.

After the extraction is completed, filter with filter paper (5.7), and then check the pH value of the extract, which shall be between 7.0 and 8.0. If it exceeds this range, it needs to reduce the mass of the sample and carry out the extraction

and the absorbance is determined by the same method, and denote it as E_2 .

7.3 Blank solution

TAKE a 25 mL volumetric flask; ADD phosphate buffer (4.2) to three-quarters of the volumetric flask; ADD 0.5 mL of phosphoric acid solution (4.4) and 0.5 mL of 1,5-diphenylcarbazide solution (4.3); USE phosphate buffer (4.2) to make the volume constant and SHAKE well. The solution shall be prepared for immediate use and placed in a dark place before use.

7.4 Plotting of standard working curve

In the range of 0.5 mL ~ 15 mL of chromium standard solution (4.7), PREPARE at least 6 standard working solutions, and PLOT a suitable standard working curve, ensuring that the range of the standard working curve is within the linear measurement range of the spectrophotometer. PIPETTE a certain amount of chromium standard solution (4.7) into 25 mL volumetric flasks; ADD 0.5 mL of phosphoric acid solution (4.4) and 0.5 mL of 1,5-diphenylcarbazide solution (4.3) to each volumetric flask; DILUTE with buffer solution (4.2) and make the volume constant; LET STAND for (15 ± 5) min.

Taking the blank solution (7.3) as a reference, use a 2 cm cuvette to determine the absorbance of the standard working solutions at 540 nm.

PLOT a standard working curve with absorbance as the Y axis and the mass concentration of chromium ($\mu\text{g/mL}$) as the X axis.

NOTE: Tests have proved that 2 cm cuvettes are the most suitable. The above standard working solutions are for testing with 2 cm cuvettes. In some cases, it may be suitable to use cuvettes with a longer or shorter optical path.

7.5 Determination of recovery rate

7.5.1 Influence of matrix

PIPETTE 10 mL of the analytical solution obtained in 7.1, ADD an appropriate amount of chromium standard solution, so that the content of chromium is close to 2 times ($\pm 25\%$) of the content of chromium in the original analytical solution (7.1). The final volume of the solution after adding the chromium standard solution does not exceed 11 mL. Then use the same method as the sample to process and determine the absorbance (see 7.2), and denote them as E_{1s} and E_{2s} respectively.

The absorbance shall be within the range of the standard working curve, otherwise it shall reduce the pipetting volume of the analytical solution and repeat the test. The recovery rate shall be greater than 80 %.

E_1 - the absorbance of analytical solution with diphenylcarbazide solution;

E_2 - the absorbance of analytical solution without diphenylcarbazide solution;

ρ - the mass concentration of the chromium added, in micrograms per milliliter ($\mu\text{g/mL}$);

F - the gradient of the standard working curve.

8.4 Expression of results

For the chromium content in the sample, it takes the arithmetic mean value of the two parallel test results as the test result, expressed in mg/kg, to the nearest 0.1 mg/kg. The ratio of the difference between the two parallel test results to the mean value shall be less than 10 %.

If the test result is based on the absolute dry mass of the sample, it shall indicate the volatile content (%) in the sample, to the nearest 0.1 %.

The detection limit of this method is 3 mg/kg.

If the detected chromium content exceeds 3 mg/kg, it shall compare the UV spectrum of the analytical solution (7.1) and the chromium standard solution (4.7), to determine whether the positive result is caused by interfering substances.

The test result of this method and the determination result of GB/T 38402 shall have a similar trend (see Annex C for the comparison of the two methods). In case of dispute, the test result of GB/T 38402 shall prevail.

9 Test report

The test report shall include the following:

- a) the series number of this Standard;
- b) sample name, type and detailed information about sampling;
- c) the type of decolorizing agent;
- d) the thickness of the cuvette, if a 2 cm cuvette is not used;
- e) the chromium content of in the sample (mg/kg), to the nearest 0.1 mg/kg;
- f) the volatile content (%) in the sample, to the nearest 0.1 %, if the result is based on the absolute dry mass of the sample;

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