NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB/T 22930.2-2021

Leather and fur - Chemical determination of metal content - Part 2: Total metal content

Issued on: October 11, 2021 **Implemented on: May 1, 2022**

Issued by: SAMR; SAC

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1 Scope

This document describes the test methods for the total contents of 24 metals: aluminum (Al), arsenic (As), barium (Ba), calcium (Ca), cadmium (Cd), cobalt (Co), chromium (Cr), copper (Cu), iron (Fe), potassium (K), magnesium (Mg), sodium (Na), lead (Pb), manganese (Mn), molybdenum (Mo), nickel (Ni), mercury (Hg), antimony (Sb), selenium (Se), silicon (Si), tin (Sn), titanium (Ti), zinc (Zn) and zirconium (Zr).

This document is applicable to the determination of the total contents of 24 metals in

various types of leather and fur, as well as the determination of non-metallic boron (B) content.

This document does not apply to the determination of total chromium content in chrome-tanned leather.

NOTE: for the determination of total chromium content in chrome-tanned leather, see QB/T 2720,

QB/T 5313, QB/T 5314 or QB/T 5315.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods (GB/T 6682-2008, ISO 3696:1987, MOD)

GB/T 39364 Leather - Chemical, Physical, Mechanical and Fastness Tests - Sampling Location (GB/T 39364-2020, ISO 2418:2017, 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 Leather - Chemical Tests - 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 1276 Fur - Chemical Tests - Determination of Matter Soluble in Carbon Tetrachloride (QB/T 1276-2012, ISO 4048:2008, 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 (QB/T 2717-2018, ISO 4684:2005, MOD)

QB/T 2718 Leather - Chemical Tests - Determination of Matter Soluble in Dichloromethane (QB/T 2718-2018, ISO 4048:2008, MOD)

3 Terms and Definitions

This document does not have terms or definitions that need to be defined.

4 Principle

Leather or fur specimen is completely digested by tribasic acid mixture (acid digestion method)

or microwave digestion apparatus (microwave digestion method). Use water to dissolve the residue, then, adopt the inductively coupled plasma optical emission spectrometer (ICP-OES), inductively coupled plasma mass spectrometer (ICP-MS), atomic absorption spectrometer (AAS) or atomic fluorescence spectrometer (AFS) to determine the metal concentration;

calculate the total metal content in the specimen.

NOTE: this document determines the total contents of metal elements in leather and fur, not the content of metal in a specific compound or oxidation state.

5.1 Unless it is otherwise specified, the chemical reagents used for the acid digestion method

are of analytical purity, and the microwave digestion method shall use acids of superior-grade purity. All used solutions are aqueous solutions.

5.3 Sulfuric acid, with a mass fraction of 98%.

8.1.1 Acid digestion method Use an analytical balance (6.3) to weigh-take 1 g of the specimen (accurate to 0.001 g) into a Kjeldahl flask (6.5); use a measuring cylinder (6.15) to add 10 mL ~ 20 mL of tribasic acid mixture (5.5); put in several glass beads (6.9). On the neck of the flask, place a funnel or water-splashing ball; heat it to boiling and maintain it in a boiling state, until the digestion is complete (the red vapor of nitrogen dioxide disappears), then, stop heating. If the specimen is not completely digested, cool the flask, then, add 10 mL ~ 20 mL of tribasic acid mixture (5.5);

repeat the above-mentioned steps.

When determining the lead and barium contents in the specimen, the digestion process shall be separately carried out.

NOTE: for the determination of high-volatile metal content, ensure that the open acid digestion process will not cause a loss of elements.

After cooling, use 30 mL of water (5.8) to dissolve the residue and transfer it to a 100 mL volumetric flask (if necessary, filter it). Use 30 mL of water to thoroughly wash the flask and

the funnel; transfer all the washing solution into a volumetric flask; use water to reach a constant volume.

Use an equivalent amount of tribasic acid mixture to replace the specimen. Then, in accordance with the above-mentioned operation, conduct a blank test.

8.1.2 Microwave digestion method The specimen digestion solution may also be prepared through the microwave digestion method or other suitable methods. If this method is used, the weighing amount of the specimen is adjusted to 0.1 g ~ 1.0 g, accurate to 0.001 g.

8.2 Instrumental Analysis

8.2.1 General rules If the content of metal elements in the obtained specimen digestion solution in 8.1 is within the range of the standard curve, it may be directly used for analysis and testing. Otherwise, the digestion solution should be properly diluted, then, tested.

The preparation of the metal standard solution to be tested and its standard working solution shall be conducted in accordance with the stipulations of Appendix C. When drawing the standard curve, make sure that the acid concentration in the metal element standard working solution is consistent with the acid concentration in the specimen digestion solution. The drawing of the standard working curve at least includes 4 series of concentrations and 1 blank concentration.

8.2.2 ICP method

8.2.2.1 ICP-OES method In accordance with the instruction manual of the instrument, set the parameters of ICP-OES (6.10). If necessary, in accordance with the instruction manual of the instrument, use a hydride generator to determine the contents of arsenic (As), antimony (Sb), tin (Sn), selenium (Se) and mercury (Hg) in the specimen.

Take the metal element solution with an already-known concentration as the reference. At the specific wavelength of each metal element, use ICP-OES to analyze the specimen digestion solution prepared in 8.1. The commonly selected determination wavelengths of the various metal elements are shown in Appendix D. In accordance with the same determination conditions as the specimen digestion solution, analyze the blank test solution.

8.2.2.2 ICP-MS method In accordance with the instruction manual of the instrument, set the parameters of ICP-MS (6.12). Take the metal element solution with an already-known concentration as the reference.

At the characteristic ion mass of each metal element, use ICP-MS to analyze the specimen digestion solution prepared in 8.1. The analytical isotopes and the method detection limits of the various metal elements are shown in Appendix E. In accordance with the same determination conditions as the specimen digestion solution, analyze the blank test solution.