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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 22931-2026

Replacing GB/T 22931-2008

Leather and fur - Chemical tests - Determination of phthalate content

皮革和毛皮 化学试验 邻苯二甲酸酯含量的测定

Issued on: May 25, 2026 **Implemented on: December 1, 2026**

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Table of Contents

Foreword

GB/T 22931-2026 | Leather and fur - Chemical tests - Determination of phthalate content

GB/T 22931-2026 English version. Leather and fur - Chemical tests - Determination of phthalate content ICS

46 National Standards of the People's Republic of China Replaces GB/T 22931-2008 Chemical testing of leather and fur Determination of Phthalate Content Published on 2026-05-

25 Implemented on December 1, 2026 State Administration for Market Regulation The State Administration for Standardization issued a statement.

1.Scope This document describes a test method for determining the phthalate content in leather and fur using solvent extraction. This document applies to the determination of phthalate content in various types of leather, fur and their products. Appendix A provides a non-solvent extraction method for determining the phthalate content in leather and fur. This document and Appendix A provide... The methods developed have similar trends, but the results are not absolutely identical. In the event of a dispute or arbitration, the results of the solvent extraction method shall prevail.

4.Principles Leather or fur samples were extracted with toluene in an ultrasonic generator, and the extract was then subjected to gas chromatography-mass spectrometry (GC-MS). The determination was performed using the internal standard method for quantification.

5 Reagents and Materials

5.1 Unless otherwise specified, all reagents used are of analytical grade.

5.2 Toluene, chromatographic grade, CAS No.. 108-88-3.

5.3 Internal standard. Any of the following substances may be used as an internal standard.

---Di(2-ethylhexyl) phthalate-3,4,5,6-D4, CAS No.. 93951-87-2;

--- Diphenyl phthalate, CAS No.. 84-62-8.

Note. When the content of di(2-ethylhexyl) phthalate (DEHP) is less than 1%, the detection of diphenyl phthalate will yield appropriate results. When the DEHP content exceeds 1%, due to the difficulty in effectively separating DEHP from diphenyl phthalate, the testing of all other phthalates is... It will be affected.

5.4 Phthalate standard samples/standard substances, see Appendix B.

5.5 Standard stock solutions. Determine the types of phthalates to be tested according to specific testing requirements. Standard stock solutions for each phthalate... The prepared solution should be a commercially available mixed standard solution, a single standard solution, or a single standard solution prepared by the laboratory using toluene. Example. Based on the purity of each phthalate (5.4), accurately weigh an appropriate amount of phthalate standard substance (5.4), dissolve and dilute it with toluene (5.2), and... Make up to volume and prepare a standard stock solution with a mass concentration of 1000 μg/mL.

5.6 Internal standard solution. Accurately weigh an appropriate amount of the internal standard (5.3), dissolve and dilute it with toluene (5.2) to prepare a solution with the same mass concentration as the standard stock solution. Internal standard solutions with the same concentration (e.g., 1000 μg/mL).

5.7 Standard working solutions. Dilute the standard stock solution (5.5) with toluene (5.2) to at least five suitable concentrations as needed. Prepare a solution and add an appropriate amount of internal standard solution (5.6). The concentration of the internal standard in each standard working solution should be the same.

5.8 Prepare an internal standard extraction solution by diluting the internal standard solution (5.6) with toluene (5.2) to achieve the same concentration of internal standard as in the standard working solution (5.7). Standard extract.

6.1 Analytical balance with a graduation of

0.1 mg.

6.2 Extraction bottle, made of glass, sealable.

6.3 Ultrasonic generator, with temperature control up to (60±5)°C.

6.4 Filter membrane, made of polytetrafluoroethylene, with a pore size of 0.45μm. 6.5-liter volumetric flasks, in various sizes.

6.6 Gas chromatograph vial, 2 mL.

6.7 Gas Chromatography-Mass Spectrometry (GC-MS).

7 Sampling and Sample Preparation

7.1 Sampling The leather shall be manufactured in accordance with the provisions of GB/T 39364. The sampling of fur shall be carried out in accordance with the provisions of QB/T 1267.The sampling process shall avoid damage to the fur and keep the fur intact. If sampling cannot be taken from a standard location (such as directly from shoes or clothing), samples should be taken from any location within the usable area. The sample should... It is representative and should be noted in the test report.

7.2 Sample Preparation The leather shall be manufactured in accordance with the provisions of QB/T 2716. The fur shall be tested in accordance with the provisions of QB/T 1272, and the sample shall be tested together with the fur. The sample size is 3mm~5mm, and the glue and adhering substances on the sample surface are removed to the greatest extent possible during the sample preparation process. To avoid cross-contamination, the cutting device should be cleaned with acetone before sample preparation.

8 Test Procedure

8.1 Preparation of Extraction Solution Accurately weigh (1.0±0.1) g of sample (accurate to 0.001

g) into extraction flask (6.2), add 10 mL of internal standard extraction solution (5.8) to moisten the sample. The sample was sealed and placed in an ultrasonic generator (6.3) at (60±5)°C for (60±5) min for extraction. After cooling to room temperature, it was filtered through a filter membrane (6.4). Transfer the filtrate to a gas analysis bottle (6.6) for later use. If the sample is not wetted, increase the amount of internal standard extract appropriately and record the actual volume added for result calculation. Toluene (5.2) can also be used for direct extraction. Before extraction, an appropriate amount of internal standard solution (5.6) should be added to the extraction flask to adjust the concentration of the internal standard. The concentration of the internal standard is the same as that in the standard working solution.

8.2 Blank solution Except for the absence of a sample, all other operations shall be performed in accordance with the provisions of 8.1.

8.3 Plotting Standard Working Curves Take appropriate amounts of standard working solutions of various concentrations and perform chromatographic analysis according to the provisions of 8.4.Plot the standard working solutions using peak areas and concentrations. curve.

8.4 GC-MS determination

8.4.1 Analysis Conditions Various types of GC-MS can be used, and the analytical conditions vary depending on the instrument. The conditions given below have been proven to be feasible.

8.4.2 Measurement The extraction solution (8.1) and blank solution (8.2) were analyzed by GC-MS according to the conditions given in 8.4.1. If the peak area in the extraction solution exceeds the range of the standard working curve, it should be appropriately diluted with internal standard extract (5.8) before proceeding. Measurement.

9 Result Calculation and Representation

9.1 Standard Operating Curve According to formula (1), the standard working curve is plotted using the ratio of (As/Ais) and (rhos/rhois).

9.2.2 Total amount of phthalates According to the agreement between the relevant parties, the test results can also be expressed as the total amount of phthalates. In this case, the total amount of phthalates included should be clearly indicated. Types of formate esters. The total amount of phthalates in the sample can be obtained by adding the test results of various phthalate contents (9.2.1). If a certain... If the phthalate test result is below the limit of quantitation of the test method, it will not be included in the total amount.

10 Feasibility of the method

10.1 Limit of Quantification The limit of quantitation (LOQ) for this method is 50 mg/kg.

10.2 Precision The ratio of the difference between two parallel test results to the average value should be less than 20%.

10.3 Recovery rate The spiked recoveries of each phthalate should be 85% to 120%.

11 Test Report The test report should include the following.

a) This document number (i.e., GB/T 22931);

b) Detailed information about the sample, including any discrepancies between the sampling method and GB/T 39364 or QB/T 1267;

c) Test method (solvent extraction or non-solvent extraction);

d) The content (mg/kg or %) of each phthalate in the sample, if on an oven-dry basis, should be clearly stated;

e) If necessary, the total amount of phthalates in the sample (mg/kg or %);

f) Abnormal phenomena observed during the experiment;

g) Any deviation from the provisions of this document.