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Leather and fur - Chemical tests - Determination of quinoline compounds

皮革和毛皮 化学试验 喹啉类化合物的测定

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Foreword

This document complies with the provisions of GB/T 1.1-2020 "Standardization Work Guidelines Part

1.Structure and Drafting Rules of Standardization Documents". Drafting. Please note that some content in this document may involve patents. The issuing organization of this document assumes no responsibility for identifying patents. This document was proposed by the China National Light Industry Council. This document is under the jurisdiction of the National Technical Committee on Standardization of Leather Industry (SAC/TC252). This document was drafted by: China Light Industry Inspection and Certification (Jinjiang) Co., Ltd., Shenzhen Institute of Metrology and Quality Inspection, and Chongqing Institute of Metrology and Quality Inspection. Testing and Research Institute, China Leather and Footwear Research Institute Co., Ltd., Kressdan (Guangdong) Holdings Co., Ltd., China Light Industry Inspection and Certification Co., Ltd., Dongguan Dongguan Weiside Technology Development Co., Ltd. The main drafters of this document are. Lin Ziwei, Chen Junrong, Cai Baixue, Gao Ya, Zhang Wenying, Wang Xichen, Gao Shenghui, Li Kehang, and Zhang Wenfu. Chemical testing of leather and fur Determination of quinoline compounds Warning

---Personnel using this document should have practical experience working in a formal laboratory. This document does not specify all possible safety precautions. All issues. Users are responsible for taking appropriate safety and health measures and ensuring compliance with relevant national regulations.

1.Scope This document describes a test method for determining the content of quinoline

compounds in leather and fur. This document applies to the determination of quinoline compounds in various types of leather, fur, and their products.

4.Principles Leather or fur samples were extracted with methanol in an ultrasonic generator. The extract was then filtered through a filter membrane and passed through a gas chromatograph-mass spectrometer. (GC-MS) determination and confirmation, external standard method for quantification.

5 Reagents and Materials

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

5.2 Methanol, chromatographic grade.

5.3 Quinoline compound standard samples/standard substances, see Appendix A.

5.4 Standard Stock Solution. Accurately weigh appropriate amounts of quinoline compound standard samples/substances (5.3), dissolve and determine their concentrations in methanol (5.2). The solution was prepared into a single-component standard stock solution of the target compound with a mass concentration of 1000 mg/L.

5.5 Mixed Standard Solutions. Transfer

1.0 mL of each standard stock solution (5.4) to the same volumetric flask, dilute with methanol (5.2), and bring to a final volume. Prepare a mixed standard solution with a mass concentration of 100 mg/L using 10 mL of the solution.

5.6 Mixed standard working solution. Dilute the mixed standard solution (5.5) with methanol (5.2) to a suitable mass concentration as needed (e.g., A series of concentrations were prepared using concentrations of

0.5 mg/L,

50.0 mg/L. A mixed standard working solution.

6 Instruments and Equipment

6.1 Gas chromatography-mass spectrometry (GC-MS) system with an electron ionization source (EI).

6.2 Analytical balance with a scale division of 0.01g.

6.3 Ultrasonic generator, with temperature control up to (60±5)°C and operating frequency of (40±5)kHz.

6.4 Reaction flasks with screw caps for airtight sealing, with a capacity of 20 mL to 40 mL.

6.5 Organic phase microporous filter membrane with a pore size of 0.45 μm.

6.6 Pipettes or pipettes, in various sizes.

7 Sampling and Sample Preparation

7.1 Sampling Leather shall be processed in accordance with GB/T 39364, and fur shall be processed in accordance with QB/T 1267. If sampling cannot be taken from a standard location (such as directly from shoes or clothing), samples can be taken from any location within the usable area. The sample should be... 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 was cut into small pieces of approximately 5mm x 5mm.

8.1 Extraction of the Sample Accurately weigh

1.0 g (accurate to

0.01

g) of the sample and place it in a reaction flask (6.4). Use a pipette or pipette (6.6) to accurately add

10.0 mL of the sample. The sample was immersed in methanol (5.2). Immediately after sealing, the reaction flask was placed in an ultrasonic generator (6.3) and extracted at (60±5)°C. 2) min. Cool to room temperature, filter through a 0.45 μm filter membrane, and use for GC-MS determination and confirmation. If the sample cannot be completely submerged in methanol, the amount of sample weighed can be appropriately reduced or the volume of methanol (5.2) can be increased, and the amount of sample weighed or the amount of methanol added should be recorded. The volume of methanol (5.2) was used for the result calculation.

8.2 Measurement

8.2.1 GC-MS Analysis Conditions Test results depend on the instrument used; therefore, it is impossible to provide universal parameters for GC-MS. The parameters set should ensure the accuracy of the chromatographic determination. The analyte can be effectively separated from other components. The parameters given below have been proven to be feasible.

a) Chromatographic column. DB-WAXUI (30mx0.250mm, 0.50μm), or equivalent.

b) Programmed temperature rise. Initial temperature 100°C, hold for 1 min; rise to 240°C at

20°C/min, hold for 2 min.

c) Carrier gas. Helium, purity >= 99.999%, flow rate

d) Inlet temperature. 250°C.

e) Injection method. split injection.

f) Injection volume. 1.0 μL.

g) Interface temperature. 280°C.

h) Ion source. EI.

i) Ionization energy. 70 eV.

j) Measurement mode. Selected ion scan (SIM), the characteristic ions of the 6 target compounds are listed in Appendix B.

k) Solvent delay time.

8.2.2 Qualitative determination Under the same experimental conditions, the target analyte in the sample solution and the simultaneously detected standard sample/standard substance have the same retention time, and The relative abundance of qualitative ions in each component of the sample spectrum is compared with the relative abundance of corresponding qualitative ions in the spectrum of the standard solution with similar concentrations. If the deviation does not exceed the range specified in Table 1, it can be determined that the corresponding target analyte is present in the sample. Under the above chromatographic conditions, the standard sample... The spectra of the standard/sample are shown in Appendix C.

8.2.3 Quantitative determination Standard curves were plotted with peak area and mass concentration of the target compound as the ordinate and abscissa, respectively, and the external standard method was used for determination. Quantitative calculation. The content of the target analyte in the sample solution should be within the range of the standard working curve. If it exceeds the range of the standard working curve, methanol should be used. (5.2) Analyze after dilution to an appropriate concentration.

9.1 Result Calculation

9.2 Results Representation The experimental results are taken as the arithmetic mean of two parallel measurements, accurate to one decimal place.

10 Feasibility of the method

10.1 Limit of Quantification The limit of quantitation for the method specified in this document is

10.2 Precision The ratio of the difference between two parallel test results to the average