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Test method for synthetic leather - Determination of short chain and medium chain chlorinated paraffins

人造革合成革试验方法 短链和中链氯化石蜡的测定

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Foreword

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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 Plastic Products (SAC/TC48). This document was drafted by: Kunshan Achilles New Materials Technology Co., Ltd., Anhui Huiyun Plastic New Materials Technology Co., Ltd., and Sichuan University. Kunshan Acas Testing Technology Service Co., Ltd., Kunshan Beice Testing Technology Co., Ltd., Shandong Tongda Island New Materials Co., Ltd. Suzhou Ruigao New Materials Co., Ltd., Chongqing Changan Automobile Co., Ltd., Shanghai Huafeng Testing Technology Co., Ltd., Nanjing University of Science and Technology Xue, Zhejiang Hexin New Materials Co., Ltd., Zhejiang Jiake New Materials Technology Co., Ltd., Fujian Zhongyu Waterborne Microfiber Technology Co., Ltd., Tianshou (Fujian) Microfiber Technology Co., Ltd., Hubei Qili Polymer Materials Co., Ltd., Fujian Huaxia Synthetic Leather Co., Ltd., Jiangxi Sangao New Materials Co., Ltd. Materials Co., Ltd., Hubei Guoyuan Waterborne New Materials Co., Ltd., Zhejiang Huafeng Synthetic Resin Co., Ltd., An'an (China) Co., Ltd. Suzhou Best Decorative New Materials Co., Ltd., Zhejiang Changfeng New Materials Co., Ltd., Chongqing Shuangxiang Microfiber Materials Co., Ltd., Shanghai Huideke Technology Co., Ltd., Dongguan Weiside Technology Development Co., Ltd., Kunshan Xiefu New Materials

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1.Scope This document describes a method for determining short-chain and medium-chain chlorinated paraffins in artificial and synthetic leather. This document applies to the determination of short-chain and medium-chain chlorinated paraffins in artificial leather, synthetic leather and their products.

4.Principles Using n-hexane as the extraction solvent, the extraction was performed by ultrasonic extraction, followed by gas chromatography-mass spectrometry (GC-MS) with an electron capture negative chemical ion source. The extract was qualitatively analyzed using GC-ECNI-MS and quantified using the internal standard method.

5 Reagents

5.1 n-Hexane n-Hexane (CAS No. 110-54-3). chromatographic grade.

5.2 Internal standard solution Hexachlorocyclohexane (CAS No. 58-89-9). 1000 μg/mL, solvent is n-hexane.

5.3 Short-chain chlorinated paraffin standard solution

5.3.1 Short-chain chlorinated paraffin standard solution with a chlorine content of 55.5%. 100 μg/mL, solvent is n-hexane.

5.3.2 Short-chain chlorinated paraffin standard solution with a chlorine content of 63.0%. 100 μg/mL, solvent is n-hexane.

5.4 Medium-chain chlorinated paraffin standard solution

5.4.1 Medium-chain chlorinated paraffin standard solution with a chlorine content of 52.0%. 100 μg/mL, solvent is n-hexane.

5.4.2 Medium-chain chlorinated paraffin standard solution with a chlorine content of 57.0%. 100 μg/mL, solvent is n-hexane.

6 Instruments

6.1 Analyzing the Balance Scale The smallest division value is 0.001g.

6.2 Glassware plug.

6.3 Ultrasonic water bath device It can maintain a temperature of (50±5)°C, and the effective ultrasonic power density ranges from 0.3W/cm2 to 2.0W/cm2.

6.4 Gas Chromatography-Mass Spectrometry System It is equipped with an electron-capturing negative chemical ion source.

7.Sampling After conditioning for at least 4 hours according to the requirements of Level 2 of Standard Environment 23/50 in GB/T 2918-2018, cut to a size less than 3mm x Prepare 3mm sample blocks, mix thoroughly, and set aside.

8.1.1 Sample Preparation and Analysis

8.1.1.1 Prepare a calibration working solution (50 μg/mL) of short-chain chlorinated paraffin with a chlorine content of 59.0%. Take 533 μL of a short-chain chlorinated paraffin standard solution (5.3.1) with a chlorine content of 55.5% and 467 μL of a short-chain chlorinated paraffin standard solution (5.3.1) with a chlorine content of 63.0%, respectively. Pour the chlorinated paraffin standard solution (5.3.2) into a 2 mL volumetric flask, add 20 μL of internal standard solution (5.2), and n-hexane (5.1). Set the appearance.

8.1.1.2 Sample Extraction Weigh (0.500±0.001) g of sample into a glass sample vial using an analytical balance (6.1), and add

9.9 mL of n-hexane (5.1) and Add 100 μL of internal standard solution (5.2), seal the glass sample vial, and shake until the sample is completely wetted. Place in an ultrasonic water bath at (50±5)°C. Extract (60±2) min in (6.3) and then cool to room temperature.

8.1.1.3 Measurement The calibration working solution and... were analyzed using a gas chromatography-mass spectrometry (GC-ECNI-MS) equipped with an electron capture negative chemical ion source (6.4). The extract was tested. Because test results depend on the instrument used, it is impossible to provide universal parameters for instrumental analysis. The parameters set should ensure that the measured... The components were effectively separated and tested. The parameters given in Table 1 have been proven feasible; adjustments can be made to the parameters for different instrument models to obtain the desired results. Optimal results.

8.1.2 Qualitative and Quantitative Analysis

8.1.2.1 Qualitative Analysis By comparing the retention time, peak shape, and... Qualitative analysis was performed using the ratio of ion abundance to qualitative and quantitative ion abundance. Appendix A provides a reference example for evaluating the peak shape of short-chain chlorinated paraffins. After confirming the presence of short-chain chlorinated paraffins in the sample, quantification was performed using the internal standard method.

8.1.2.2 Quantitative Analysis The sum of the quantitative ion peak areas in the calibration working solution of short-chain chlorinated paraffin with a chlorine content of 59.0% was

used to establish a calibration method with respect to the mass concentration. The curve was used to perform quantitative calculations on the sample extraction solution using the internal standard method. The total ion chromatogram (TIC chromatogram) of the sample extraction solution should be obtained from the abundance of the calibrated working solution containing short-chain chlorinated paraffin with a chlorine content of 59.0%. If the sample is outside the specified range, the sample extraction solution should be diluted and the test repeated. The quantitative ion abundance ratio of the sample and the quantitative ion abundance ratio of the calibration working solution of short-chain chlorinated paraffin with a chlorine content of 59.0% should be controlled. The deviation is within ±30%. A short-chain chlorinated paraffin calibration working solution with a chlorine content of 59.0% was added at the end of the sequence or after up to 10 samples as a quality control. For sample preparation and quality control, the test results deviation should be within ±20%. If it exceeds this range, the calibration curve should be re-established and the test should be repeated. The new batch of samples was tested.

8.1.2.3 Interference When the content of medium-chain chlorinated paraffins is high, it can interfere with the quantitative determination of C12Cl7 ions (m/z 375). In this case, interchangeable determination methods can be used. The quantitative and qualitative ions should be identified and noted in the test report.

8.1.2.4 Calculation A linear regression function is established using the ratios (ASCCP-S/Aint.Std-S) and (rhoSCCP-S/rhoint.Std-S), as shown in formula (1).

8.2.1 Sample Preparation and Analysis

8.2.1.1 Prepare a calibration working solution (50 μg/mL) of medium-chain chlorinated paraffin with a chlorine content of 55.0%. Take 400 μL of a medium-chain chlorinated paraffin standard solution with a chlorine content of 52.0% (5.4.1) and 600 μL of a medium-chain chlorinated paraffin standard solution with a chlorine content of 57.0%, respectively. Pour the chlorinated paraffin standard solution (5.4.2) into a 2 mL volumetric flask, add 20 μL of internal standard solution (5.2), and n-hexane (5.1). Set the appearance.

8.2.1.2 Extraction Weigh (0.500±0.001) g of sample into a glass sample vial using an analytical balance (6.1), and add

9.9 mL of n-hexane (5.1) and Add 100 μL of internal standard solution (5.2), seal the glass sample vial, and shake until the sample is completely wetted. Place in an ultrasonic water bath at (50±5)°C. Extract (60±2) min in (6.3) and then cool to room temperature.

8.2.1.3 Measurement The calibration working solution and... were analyzed using a gas chromatography-mass spectrometry (GC-ECNI-MS) equipped with an electron capture negative chemical ion source (6.4). The extract was tested. Because test results depend on the instrument used, it is impossible to provide universal parameters for instrumental analysis. The parameters set should ensure that the measured... The components were