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Determination of hexamethylenetetramine in cosmetics - Liquid chromatography-tandem mass spectrometry

化妆品中乌洛托品的测定 液相色谱-串联质谱法

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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 Fragrance, Flavor and Cosmetic Products (SAC/TC257).

This document was drafted by: Suzhou Shipu Testing Technology Co., Ltd., Suzhou Kenairui Testing Technology Co., Ltd., and Suzhou Keshipu Biotechnology Co., Ltd.

Tech (Suzhou) Co., Ltd., Suzhou Quality Inspection and Science Research Institute, Perfect (Guangdong) Daily Necessities Co., Ltd., Guangzhou Quality Supervision and Inspection Institute Osmun Biotechnology Co., Ltd., Guangzhou Mashanghui Biotechnology Co., Ltd., Proya Cosmetics Co., Ltd., Shanghai Fragrance Research Institute Limited Liability Company, Jiangsu Provincial Institute of Product Quality Supervision and Inspection, Jiangsu Provincial Institute of Drug Supervision and Inspection, Hebei Provincial Institute of Food Inspection, Jiangsu Provincial Institute of Metrology, Guangzhou Huaxin Testing Technology Co., Ltd., Suzhou Drug Inspection and Testing Research Center, Suzhou Industrial Park Pharmaceutical Group Drug Management Center (Suzhou Branch of

Jiangsu Provincial Drug Administration Review and Inspection Center), Jilin Chemical Engineering College, Hainan University, Suzhou Customs Comprehensive Technology Technology Center, Suzhou Wujiang District Food and Drug Administration, China Pharmaceutical University, Soochow University, Tsinghua University, Shanghai Qiran Biotechnology Co., Ltd.

Company. Shanghai Xiaochuang Testing Technology Co., Ltd.

The main drafters of this document are: Liao Huayong, Chu Kunqi, Zhang Kefeng, Pan Kai, Lu Yi, Jiang Lei, Xu Zhendong, He Shuang, Lu Jian, Huang Ruijuan, and Yang Anquan.

Zhang Yan, Dai Dan, Wang Ting, Jie Li, Ye Zhuhong, Chen Laicheng, Fang Fang, Zhu Dandan, Zhang Lihua, Yang Bin, Li Jianxiang, Liu Danyang, Sun Shurong, Gao Long Zheng Jinrong, Du Xinfeng, Kang Wei, Zhao Weijun, Wang Yi, Wang Yang, Xun Zhiqing, Huang Jinfeng, Guo Xindong, Gu Pan, Lu Yinghong, Liu Qunlin, Shao Ping, Yuan Minjia, Li Qi, Wang Ying, Wu Meifang.

Introduction

The substances tested in this document are prohibited ingredients as stipulated in China's "Cosmetic Safety Technical Specifications (2015 Edition)".

Prohibited ingredients refer to substances that are not permitted to be used as ingredients in cosmetics. The "Cosmetic Safety Technical Specifications (2015 Edition)" stipulates that "if the technology..." When it is unavoidable that prohibited substances may be introduced into cosmetics as impurities, those with national limits should comply with those limits; if no limits are specified, further investigation is required.

A safety risk assessment is conducted to ensure that no harm to human health will occur under normal, reasonable, and foreseeable conditions of use.

Currently, China has not set a limit for this substance. This document is only intended to provide a detection method for determining this substance in cosmetics.

Determination of urotropine in cosmetics Liquid chromatography-tandem mass spectrometry Warning---Personnel using this document should have practical experience working in a formal laboratory. This document does not address all possible safety issues.

Users are responsible for taking appropriate safety and health measures and ensuring compliance with relevant national regulations.

1 Scope

This document describes the principle, reagents and materials, instruments and equipment, and reagents for the determination of urotropine in cosmetics by liquid chromatography-tandem mass spectrometry.

The test includes sample preparation and testing procedures, data processing, recovery rate, precision, and test report.

This document applies to the determination of urotropine in aqueous, cream, and lotion cosmetics.

The limit of detection for this method is 0.10 mg/kg, and the limit of quantitation is 0.20 mg/kg.

2 Normative references

GB/T 6682

3 Terms and Definitions

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

4 Principles

The sample was subjected to vortexing with 20% acetonitrile solution, ultrasonic extraction, purification by n-hexane liquid-liquid extraction, centrifugation, and filtration to obtain the target analyte.

Liquid. The target analyte solution was injected into a liquid chromatography-tandem mass spectrometer (LC-MS/MS) for separation by liquid chromatography and detection by mass spectrometry. Retention time and mass spectrometry were then determined.

Qualitative analysis was performed using the relative ion abundance of the spectrum, and quantitative analysis was performed using the internal standard method.

5 Reagents and Materials

Unless otherwise specified, use only chromatographically pure reagents.

5.1 Water. GB/T 6682, Grade 1. 5.2 Acetonitrile. 5.3 Ammonium acetate. 5.4 n-Hexane.

5.5 20% acetonitrile solution. Take 20 mL of acetonitrile (5.2), dilute with water (5.1) to 100 mL, and mix well.

5.6 Ammonium acetate solution (c=0.005mol/L). Weigh 0.3854g of ammonium acetate (5.3), dissolve and dilute with water (5.1) to 1L, and mix well.

5.7 Urotropine Standard Samples/References. Purity not less than 99%, English name, CAS number, and molecular weight of the standard sample/reference.

The formula, relative molecular mass and chemical structural formula are shown in Table A.1 of Appendix A.

5.8 Urotropine Internal Standard (Urotropine-D12). Purity not less than 99%, English name, CAS number, molecular formula, relative purity of urotropine-D12.

Molecular mass and chemical structural formula are shown in Table A.1.

5.9 Standard stock solution (rho=1000μg/mL). Accurately weigh 0.01g of urotropine standard sample/standard substance (5.7) (accurate to 0.0001g).

Place in a 10 mL brown volumetric flask, dissolve in acetonitrile (5.2), and dilute to the mark to obtain a standard stock with a mass concentration of 1000 μg/mL.

The liquid can be stored at -20°C or below, protected from light, for up to one month.

5.10 Internal Standard Intermediate Solution (rho=5.0μg/mL). Accurately weigh 0.01g of urotropine internal standard (5.8) (accurate to 0.0001g), and place it in a container...

In a 10 mL brown volumetric flask, dissolve and dilute to the mark with acetonitrile (5.2) to obtain an internal standard stock solution with a mass concentration of 1000 μg/mL.

Store at -20°C or below, protected from light, for up to one month. Accurately transfer 0.050 mL of the internal standard stock solution into a 10 mL amber volumetric flask.

Dilute to the mark with acetonitrile (5.2) to obtain an internal standard intermediate solution with a mass concentration of 5.0 μg/mL. Prepare and use immediately.

5.11 Filter membrane. pore size 0.22μm, material is polytetrafluoroethylene (PTFE).

6 Instruments and Equipment

6.1 Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS). Equipped with an electrospray ionization source.

6.2 Analytical balance. with an accuracy of 0.001g and 0.00001g. 6.3 Vortex Oscillator.

6.4 Ultrasonic cleaner. operating frequency not less than 40kHz.

6.5 Centrifuge. Maximum speed not less than 4000 r/min.

7.1 Sample Preparation

Before sampling, the sample should be thoroughly mixed. For aqueous cosmetics, shake the sample well before sampling; for creams and lotions, squeeze out the sample... Take a sample after mixing.

Weigh 0.5 g (accurate to 0.001 g) of the sample and place it in a 25 mL stoppered colorimetric tube. Accurately add 0.400 mL of internal standard intermediate solution.

(5.10) Vortex mix thoroughly. Accurately add 8 mL of 20% acetonitrile (5.5), stopper and vortex mix thoroughly, sonicate for 10 min, and cool to room temperature.