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**Determination of acrylamide in toothpaste - High performance
liquid chromatography tandem mass spectrometry**

牙膏中丙烯酰胺的测定 高效液相色谱串联质谱法

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Foreword

This document is in accordance with the provisions of GB/T 1.1-2020 "Guidelines for standardization work Part

1. Structure and drafting rules for standardization documents" Drafting is required. Please note that some of the contents of this document may involve patents. The issuing organization of this document does not assume the responsibility for identifying patents. This document was proposed by China Light Industry Federation. This document is under the jurisdiction of the National Technical Committee for Standardization of Oral Care Products (SAC/TC 492). This document was drafted by: Suzhou Shipu Testing Technology Co., Ltd., Sukoshipu Biotechnology (Suzhou) Co., Ltd., Suzhou Quality Inspection Institute of Testing Science, Suzhou Kernairui Testing Technology Co., Ltd., Guangzhou Shuke Industrial Co., Ltd., Guangzhou Quality Supervision and Inspection Institute, Heilongjiang Light Industry Science Research Institute, Infinitus (China) Co., Ltd., Fujian Aijieli Daily Chemical Co., Ltd., Liuzhou Liangmianzhen Co., Ltd. Company, Chongqing Dengkang Oral Care Products Co., Ltd., Zhejiang Fangyuan Testing Group Co., Ltd., Hebei Food Inspection

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Determination of acrylamide in toothpaste by HPLC Tandem mass spectrometry WARNING - Personnel using this document should have practical experience in formal laboratory work. This document does not purport to address all possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring that the conditions are in compliance with relevant national regulations.

1 Scope

China's national method for determining acrylamide in toothpaste by liquid chromatography tandem mass spectrometry. Acrylamide is a neurotoxin and a probable carcinogen, and its presence in toothpaste is a residue rather than an ingredient: polyacrylamide and its derivatives are used as thickeners and binders in cosmetic formulations, and the polymer carries a small quantity of unreacted monomer. Cosmetic regulations therefore limit the residual monomer, and toothpaste attracts particular attention because it is used twice a day, is held in the mouth against the most absorptive tissue in the body, and is partly swallowed - by children, substantially so. The analysis has to reach concentrations of a few micrograms per kilogram in a matrix full of abrasives, surfactants and humectants, which is why the tandem mass spectrometry is required rather than a simpler chromatographic detection.

This document describes the principle, reagents and materials, instrumentation, and methods for the determination of acrylamide in toothpaste by high performance liquid chromatography-tandem mass spectrometry. Contents include test steps, result calculation, recovery rate and precision, allowable difference, etc. This document is applicable to the determination of acrylamide in toothpaste.

2 Normative references

The contents of the following documents constitute the essential clauses of this document through normative references in this document. For referenced documents without a date, only the version corresponding to that date applies to this document; for referenced

documents without a date, the latest version (including all amendments) applies. in this document.

GB/T 6682 Specifications and test methods for water used in analytical laboratories

3 Terms and definitions

There are no terms or definitions that require definition in this document.

4 Principle

Acrylamide in toothpaste was extracted with water and purified with n-hexane, and then detected by high performance liquid chromatography-tandem mass spectrometry in multiple reaction monitoring mode. Retention time and characteristic fragment ion abundance ratio were used for qualitative analysis, and internal standard method was used for quantitative analysis.

5 Reagents and Materials

5.1 Unless otherwise specified, all reagents used are of chromatographic grade and water is first-grade water that meets the requirements of GB/T 6682.

5.2 Acetonitrile.

5.4 Formic acid. 5.5 0.1% (volume fraction) formic acid solution. Take 1 mL of formic acid (5.4), dilute to 1 L with water, and mix well.

5.6 Standard substance/standard sample. English name, CAS number, molecular formula, relative molecular mass of acrylamide standard substance/standard sample The chemical structure is shown in Table A.1 in Appendix A, and the purity is not less than 98%.

5.7 Isotope internal standard. $^{13}\text{C}_3$ The English name, CAS number, molecular formula, relative molecular mass and chemical structure of acrylamide are shown in Table A.1. The purity is not less than 98%.

5.8 Standard stock solution. Accurately weigh 10 mg (accurate to 0.000 1

g) of acrylamide standard substance/standard sample (5.6) in a 10 mL container. Place in a volumetric flask, dissolve with acetonitrile (5.2) and dilute to the mark to obtain a standard stock solution with a mass concentration of 1 000 $\mu\text{g/mL}$. Store away from light, can be stored for 12 months.