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SINGAPORE STANDARD

Cosmetics – Analytical methods – Nitrosamines: Detection and determination of *N*-nitrosodiethanolamine (NDELA) in cosmetics by HPLC, post-column photolysis and derivatisation

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National Foreword

This Singapore Standard was prepared by the National Mirror Working Group on Cosmetics set up by the Technical Committee on Complimentary Medicine and Health Products under the purview of the Biomedical and Health Standards Committee.

This standard is an identical adoption of ISO 10130:2017, “Cosmetics – Analytical methods – Nitrosamines: Detection and determination of *N*-nitrosodiethanolamine (NDELA) in cosmetics by HPLC, post-column photolysis and derivatization”, published by the International Organization for Standardization.

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INTERNATIONAL STANDARD

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Cosmetics — Analytical methods — Nitrosamines: Detection and determination of *N*-nitrosodiethanolamine (NDELA) in cosmetics by HPLC, post-column photolysis and derivatization

Cosmétiques — Méthodes analytiques — Nitrosamines: Recherche et dosage de la N-*nitrosodiéthanolamine (NDELA) dans les cosmétiques par CLHP, photolyse et dérivation post-colonne*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 10130 was prepared by Technical Committee ISO/TC 217, *Cosmetics*.

Introduction

Human exposure to *N*-nitrosamines can occur through diverse sources such as the environment, food or personal care products. As a result of their perceived carcinogenic potential on several animal species, minimization of exposure to *N*-nitrosamines is recognized as important to the preservation of human health. Among *N*-nitrosamines, *N*-nitrosodiethanolamine (NDELA) has been recognized as a potential contaminant of cosmetics.

In this context, several analytical methods have been developed to detect and determine the presence of NDELA in cosmetics. Examples of these methods are gas chromatography/thermal energy analysis, and high performance liquid chromatography coupled either with a mass spectrometry determination or with photolysis and colorimetric quantification. The latter method uses specific technology to ensure specificity towards NDELA, to minimize the risk of artefactual formation of the analyte of interest and to allow precise quantification.

This analytical method uses High Performance Liquid Chromatography (HPLC) coupled with post-column photolysis and derivatization, in order to separate and detect trace levels of NDELA from a cosmetic ingredient or product matrix with specificity for NDELA.

This International Standard refers to a collaborative study (Reference [2]) involving seven laboratories and published in 2006. Validation criteria are given in Reference [2].

INTERNATIONAL STANDARD **ISO 10130:2009(E)**

Cosmetics — Analytical methods — Nitrosamines: Detection and determination of *N*-nitrosodiethanolamine (NDELA) in cosmetics by HPLC, post-column photolysis and derivatization

1 Scope

This International Standard describes a method for the detection and quantification of *N*-nitrosodiethanolamine (NDELA) in cosmetics and raw materials used in cosmetics by high performance liquid chromatography (HPLC) coupled with post-column photolysis and derivatization.

This method is not applicable to the detection and/or quantification of nitrosamines other than NDELA, nor to the detection and/or quantification of NDELA in products other than cosmetics or raw materials used in cosmetics.

If a product has the possibility of either NDELA contamination from the ingredients or NDELA formation by the composition of ingredients, the method will be applied for the testing of cosmetic products and is an alternative to ISO 15819.

This method is not applicable to matrices containing oxidation dyes.

2 Principle

Extraction of the nitrosamine NDELA from cosmetic samples is carried out with water. Clean-up is performed either using solid phase extraction (SPE clean-up, see 5.3.2) with a C18 cartridge or dichloromethane (DCM clean-up, see 5.3.3) when the samples are not dispersible in water. The extracts are analysed by HPLC, post-column photolysis and derivatization. NDELA is separated from the cosmetic matrix using reversed-phase liquid chromatography. The *N*-nitroso bond is cleaved by UV photolysis with the formation of nitrite ion. According to the Griess reaction, the nitrite functional group is diazotized with sulfanilamide in an acid medium and is then coupled with *N*-(1-naphthyl)ethylenediamine dihydrochloride (NED) to form a purple-coloured azo dye that is quantitatively determined spectrophotometrically at a maximum wavelength, λ_{max}, of 540 nm (see Annex B).

The presence of NDELA can be confirmed by repeating the analysis without photolysis (no nitrite ion is then produced because the *N*-nitroso bond is not cleaved). The absence of a chromatographic peak at the retention time of NDELA in the chromatogram confirms that the peak observed in the first analysis corresponds to NDELA.