

SINGAPORE STANDARD

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



Published by

Enterprise
Singapore

SS ISO 10130 : 2017

ISO 10130:2009, IDT
(ICS 71.100.70)

SINGAPORE STANDARD

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

All rights reserved. Unless otherwise specified, no part of this Singapore Standard may be reproduced or utilised in any form or by any means, electronic or mechanical, including photocopying and microfilming, without permission in writing from Enterprise Singapore. Request for permission can be sent to: standards@enterprisesg.gov.sg.

© ISO 2009 – All rights reserved

© Enterprise Singapore 2017

ISBN 978-981-47-8404-7

SS ISO 10130 : 2017

This Singapore Standard was approved by the Biomedical Standards Committee on behalf of the Singapore Standards Council on 28 April 2017.

First published, 2017

The Biomedical Standards Committee, appointed by the Standards Council, consists of the following members:

	Name	Capacity
Acting Chairman	: Mr Foo Yang Tong	<i>Individual Capacity</i>
Advisor	Ms Jacqueline Monteiro	<i>Individual Capacity</i>
Secretary	: Mr Choi Kwok Keong	<i>Singapore Manufacturing Federation – Standards Development Organisation</i>
Members	: Mr David Barda	<i>Exploit Technologies Pte Ltd</i>
	Prof Kishore Bhakoo	<i>Singapore Bioimaging Consortium</i>
	Ms Sheryl Chen	<i>Economic Development Board</i>
	Mr Chung Kwong Yuew	<i>Temasek Polytechnic</i>
	Ms Farah Binte Mohamed Haniff	<i>National Healthcare Group Pte Ltd</i>
	Prof James Goh	<i>Biomedical Engineering Society (Singapore)</i>
	Dr Stuart Koe	<i>Singapore Manufacturing Federation</i>
	Ms Evelyn Koh	<i>Ministry of Manpower</i>
	Mr Kwan Fook Weng	<i>Singapore Health Services Pte Ltd</i>
	Ms Kwek Puay Ee	<i>Singapore Nursing Board</i>
	Assoc Prof Leo Hwa Liang	<i>National University of Singapore (Biomedical Engineering)</i>
	Dr Leonard Loh	<i>Nanyang Polytechnic</i>
	Assoc Prof Eddie Ng Yin Kwee	<i>Nanyang Technological University</i>
	Dr Ong Siew Hwa	<i>Individual Capacity</i>
	Mr Justin Phoon	<i>Biomedical Research Council</i>
	Mr Poo Yi Hong	<i>International Enterprise Singapore</i>
	Ms Celine Tan	<i>SPRING Singapore</i>
	Ms Jocelyn Yen	<i>Singapore Manufacturing Federation</i>
	Dr Yong Chern Chet	<i>Individual Capacity</i>

The Working Group on Cosmetics, appointed by the Biomedical Standards Committee to assist in the preparation of this standard, comprises the following experts who contribute in their *individual capacity*:

	Name
Convenor	: Dr Alain Khaiat
Secretary	: Ms Cynthia Toh Sook Ai
Members	: Dr Maria Antipina
	Ms Stephanie Chan
	Dr Cheah Nuan Ping
	Ms Innocentia M Krisnawati

Members : Dr Khoo Keng Meng
Mr Lam Kok Seng
Dr Celine Valeria Liew
Mr Mohanram Subramaniam
Mr Pang Tit Keong
Ms Josephine Song
Mrs Marie Tham
Mr Gary Yao

The organisations in which the experts of the Working Group are involved are:

Arch Chemicals Singapore Pte Ltd
Celblos Dermal Research Centre Pte Ltd
Cosmetics, Toiletry and Fragrance Association of Singapore
Health Sciences Authority
Institute of Materials Research and Engineering
Johnson and Johnson Pte Ltd
Lubrizol Southeast Asia Pte Ltd
National University of Singapore
Procter & Gamble (S) Pte Ltd
SC Solution Pte Ltd
Singapore Polytechnic

(blank page)

Contents	Page
National Foreword	6
Foreword	7
Introduction	8
1 Scope	9
2 Principle	9
3 Reagents	9
4 Apparatus	10
5 Sample preparation and storage	11
5.1 General	11
5.2 Standards preparation	12
5.2.1 Primary stock solution	12
5.2.2 Secondary stock solution	12
5.2.3 Working solutions	12
5.3 Sample preparation	13
5.3.1 General	13
5.3.2 SPE clean-up	13
5.3.3 Alternative sample preparation for samples non-dispersible in water (DCM clean-up)	13
6 Procedure	13
6.1 General	13
6.2 Chromatographic conditions	13
6.3 Set-up of the reaction system	14
7 Calculation of results	14
7.1 Calibration curve	14
7.2 Experimental conditions for the validity of the measurement	15
7.3 Calculation of concentrations	15
8 Test report	16
Annex A (informative) Examples of calibration curves and typical chromatograms	17
Annex B (normative) Photolysis and reaction of nitrite with Griess reagent to form the azo dye	20
Annex C (normative) Configuration of the post-column reactor system	21
Bibliography	22

National Foreword

This Singapore Standard was prepared by the National Mirror Working Group on Cosmetics under the direction of the Biomedical Standards Committee. This Singapore Standard is an identical adoption of International Standard ISO 10130:2009 “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.

Attention is drawn to the following:

1. Where appropriate, the words ‘International Standard’ shall be read as ‘Singapore Standard’. The reference to ‘ISO 15819’ shall be replaced by ‘SS ISO 15819’.
2. The comma has been used throughout as a decimal marker whereas in Singapore Standards it is a practice to use a full point on the baseline as the decimal marker.

Attention is drawn to the possibility that some of the elements of this Singapore Standard may be the subject of patent rights. Enterprise Singapore shall not be held responsible for identifying any or all of such patent rights.

NOTE

1. *Singapore Standards (SSs) and Technical References (TRs) are reviewed periodically to keep abreast of technical changes, technological developments and industry practices. The changes are documented through the issue of either amendments or revisions.*
2. *An SS or TR is voluntary in nature except when it is made mandatory by a regulatory authority. It can also be cited in contracts making its application a business necessity. Users are advised to assess and determine whether the SS or TR is suitable for their intended use or purpose. If required, they should refer to the relevant professionals or experts for advice on the use of the document. Enterprise Singapore shall not be liable for any damages whether directly or indirectly suffered by anyone or any organisation as a result of the use of any SS or TR.*
3. *Compliance with a SS or TR does not exempt users from any legal obligations.*

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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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].

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.