

Methods for analysis of allergens - Quantification of suspected fragrance allergens in consumer products - Step 1: GC analysis of ready-to-inject sample

This document is a preview generated by EVS

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN 16274:2012 sisaldab Euroopa standardi EN 16274:2012 ingliskeelset teksti.	This Estonian standard EVS-EN 16274:2012 consists of the English text of the European standard EN 16274:2012.
Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas.	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 12.09.2012.	Date of Availability of the European standard is 12.09.2012.
Standard on kättesaadav Eesti Standardikeskusest.	The standard is available from the Estonian Centre for Standardisation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile standardiosakond@evs.ee.

ICS 71.100.60

Standardite reprodutseerimise ja levitamise õigus kuulub Eesti Standardikeskusele

Andmete paljundamine, taastekitamine, kopeerimine, salvestamine elektroonsesse süsteemi või edastamine ükskõik millises vormis või millisel teel ilma Eesti Standardikeskuse kirjaliku loata on keelatud.

Kui Teil on küsimusi standardite autorikaitse kohta, võtke palun ühendust Eesti Standardikeskusega:
Aru 10, 10317 Tallinn, Eesti; www.evs.ee; telefon 605 5050; e-post info@evs.ee

The right to reproduce and distribute standards belongs to the Estonian Centre for Standardisation

No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying, without a written permission from the Estonian Centre for Standardisation.

If you have any questions about copyright, please contact Estonian Centre for Standardisation:
Aru 10, 10317 Tallinn, Estonia; www.evs.ee; phone 605 5050; e-mail info@evs.ee

ICS 71.100.60

English Version

Methods for analysis of allergens - Quantification of suspected fragrance allergens in consumer products - Step 1: GC analysis of ready-to-inject sample

Méthodes d'analyse des allergènes - Quantification des fragrances allergènes suspectées dans les produits de consommation - Étape 1 : Analyse par GC d'échantillons prêts à être injectés

Analyseverfahren für Allergene - Quantifizierung von mutmaßlichen Allergie auslösenden Duftstoffen in Verbrauchsgütern - Stufe 1: GC-Analyse von einspritzfertigen Proben

This European Standard was approved by CEN on 4 August 2012.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents

Page

Foreword.....	3
Introduction	4
1 Scope	5
2 Principle	5
3 Reagents	5
3.1 Solvents	5
3.2 Fragrance (suspected allergen) standards	5
3.3 Internal standards (ISTD)	7
4 Apparatus	7
4.1 Analytical Balance	7
4.2 GC-FID (for solvent or standard purity only)	7
4.3 GC-MS	7
4.4 GC capillary columns	7
5 Standard preparation and preservation	8
5.1 General.....	8
5.2 Standard preparation	8
5.2.1 General.....	8
5.2.2 Stock solution of all allergens (5 g/l)	8
5.2.3 Separate stock solutions (carbonyl / non carbonyl compounds) (10 g/l).....	9
5.2.4 Internal standard solution.....	9
5.2.5 Working solutions.....	9
5.2.6 Calibration solution	9
6 Procedure	10
6.1 General.....	10
6.2 Chromatographic conditions.....	10
6.3 MS conditions	10
6.3.1 General.....	10
6.3.2 SCAN mode	10
6.3.3 SIM mode	11
6.4 Calibration	13
7 Sample analysis.....	13
8 Data treatment and calculation of results	14
8.1 Identification of allergens	14
8.2 Quantification of allergens	14
8.3 Assessment of the analytical measurement.....	15
8.3.1 General.....	15
8.3.2 Examination of the Q-values	15
8.3.3 Maximum permitted tolerances.....	15
9 Test report	16
Annex A (informative) Column performances	17
Annex B (informative) SIM windows.....	19
Annex C (informative) Example of chromatographic separation	20
Annex D (normative) Decisional tree	22
Bibliography	23

Foreword

This document (EN 16274:2012) has been prepared by Technical Committee CEN/TC 347 "Methods for analysis of allergens", the secretariat of which is held by DS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2013, and conflicting national standards shall be withdrawn at the latest by March 2013.

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

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

Human skin exposure to suspected allergenic fragrances can occur through diverse sources such as detergents and cosmetics intended to be rinsed or not. As a result of their possible effect, 26 fragrance substances have been restricted under Council Directives with labelling requirements in order to insure a high level of protection of consumers, particularly for sensitive population.

In this context, several analytical methods have been developed to detect and determine their presence in cosmetics such as Gas Chromatography/Flame Ionisation Detector (GC-FID), Gas Chromatography/Mass Spectrometry (GC-MS), comprehensive GC or MS-MS in raw materials and finished products.

The present analytical method uses GC-MS by combination of two GC columns of different polarity with a dedicated methodology for quantification [1]. This allows separation and quantification of the 24 volatile suspected allergens above 0,001 % (10 mg/kg) of each, in ready-to-inject sample from a cosmetic ingredient or product matrix. The present protocol has been validated thanks to a ring test [2].

1 Scope

This European Standard describes a method for the identification and determination of 24 volatile suspected allergens from ready-to-inject cosmetics and raw materials used in cosmetic products and are compatible with GC analysis. This analysis uses GC-MS after sample preparation. The 24 suspected allergens are restricted under Council Directives (7th amendment to the Cosmetic Directive 2003/15/EC).

The method described in this European Standard does not include requirements for the preparation of samples in matrices for which direct injection in GC is not feasible.

2 Principle

The method described in this European Standard is a comprehensive analysis of 24 volatile suspected allergens by Gas Chromatography coupled with Mass Spectrometry after dilution of the sample in an inert solvent.

Two assays are performed for the chromatographic separation of the 24 suspected allergens using two GC capillary columns of different polarities. Suspected allergen identification is achieved when possible using GC-MS in scan mode. Quantification is performed by single ion monitoring (SIM) using 1,4-dibromobenzene and 4,4'-dibromobiphenyl as internal standards.

The final result depends on the agreement of the different ion ratios obtained for both injections according to specific requirements.

3 Reagents

Unless otherwise stated, use only reagents of recognised analytical grade. The solvent shall be of quality for GC-MS analysis.

3.1 Solvents

3.1.1 Methyl pivalate, CAS no: [598-98-1], analytical grade or higher.

3.1.2 Ortho-fluorotoluene, CAS no: [95-52-3], analytical grade or higher.

3.1.3 Acetone, CAS no: [67-64-1], analytical grade or higher.

IMPORTANT — if other solvents are used, their inertness with the analytes shall be demonstrated. In any case, the same solvent shall be used both for calibration and determination.

3.2 Fragrance (suspected allergen) standards

3.2.1 Amylcinnamic alcohol, CAS no: [101-85-9], with known purity.

NOTE Possibly two isomers.

3.2.2 Amylcinnamic aldehyde (flosal®), CAS no: [122-40-7], with known purity.

NOTE Possibly two isomers.

3.2.3 Anisyl alcohol, CAS no: [105-13-5], with known purity.

3.2.4 Benzyl alcohol, CAS no: [100-51-6], with known purity.

3.2.5 Benzyl benzoate, CAS no: [120-51-4], with known purity.