

# norm

# NEN-EN 16344

Cosmetics - Analysis of cosmetic preparations - Detection and quantitative determination of UV filters in cosmetic preparations, HPLC method

Publicatie uitsluitend voor commentaar

november 2011  
ICS 71.100.70

Commentaar vóór 2012-03-03

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Voorbeeld  
Preview

November 2011

ICS 71.100.70

English Version

**Cosmetics - Analysis of cosmetic preparations - Detection and quantitative determination of UV filters in cosmetic preparations, HPLC method**

Cosmétiques - Analyse des préparations cosmétiques - Détection et détermination quantitative des filtres UV dans les préparations cosmétiques, méthode CLHP

Kosmetische Mittel - Untersuchung von Sonnenschutzmitteln - Screening und quantitative Bestimmung von 10 UV-Filtern in Sonnenschutzmitteln, HPLC-Verfahren

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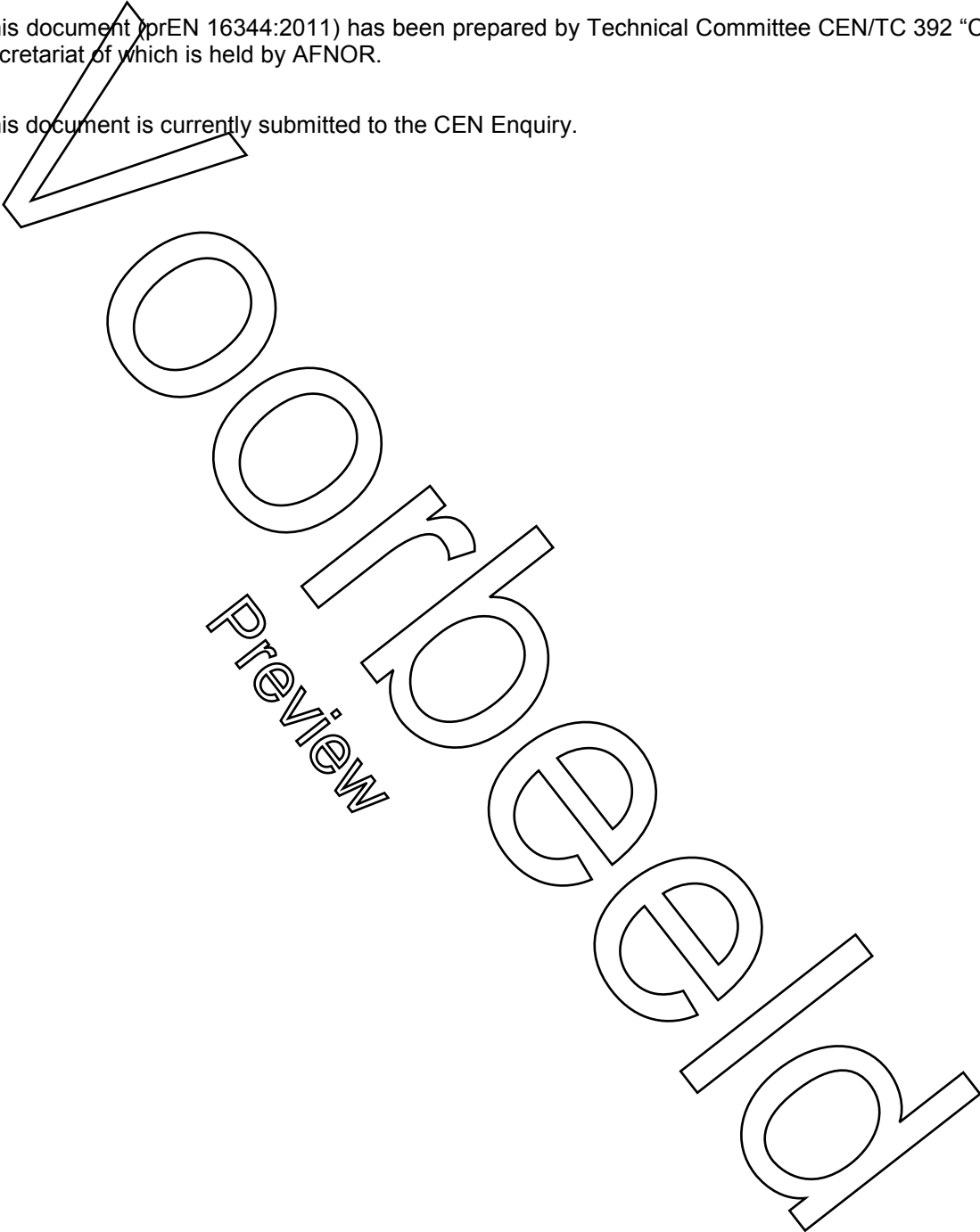
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Preview  
 NEN-EN 16344:2011

## Foreword

This document (prEN 16344:2011) has been prepared by Technical Committee CEN/TC 392 "Cosmetics", the secretariat of which is held by AFNOR.

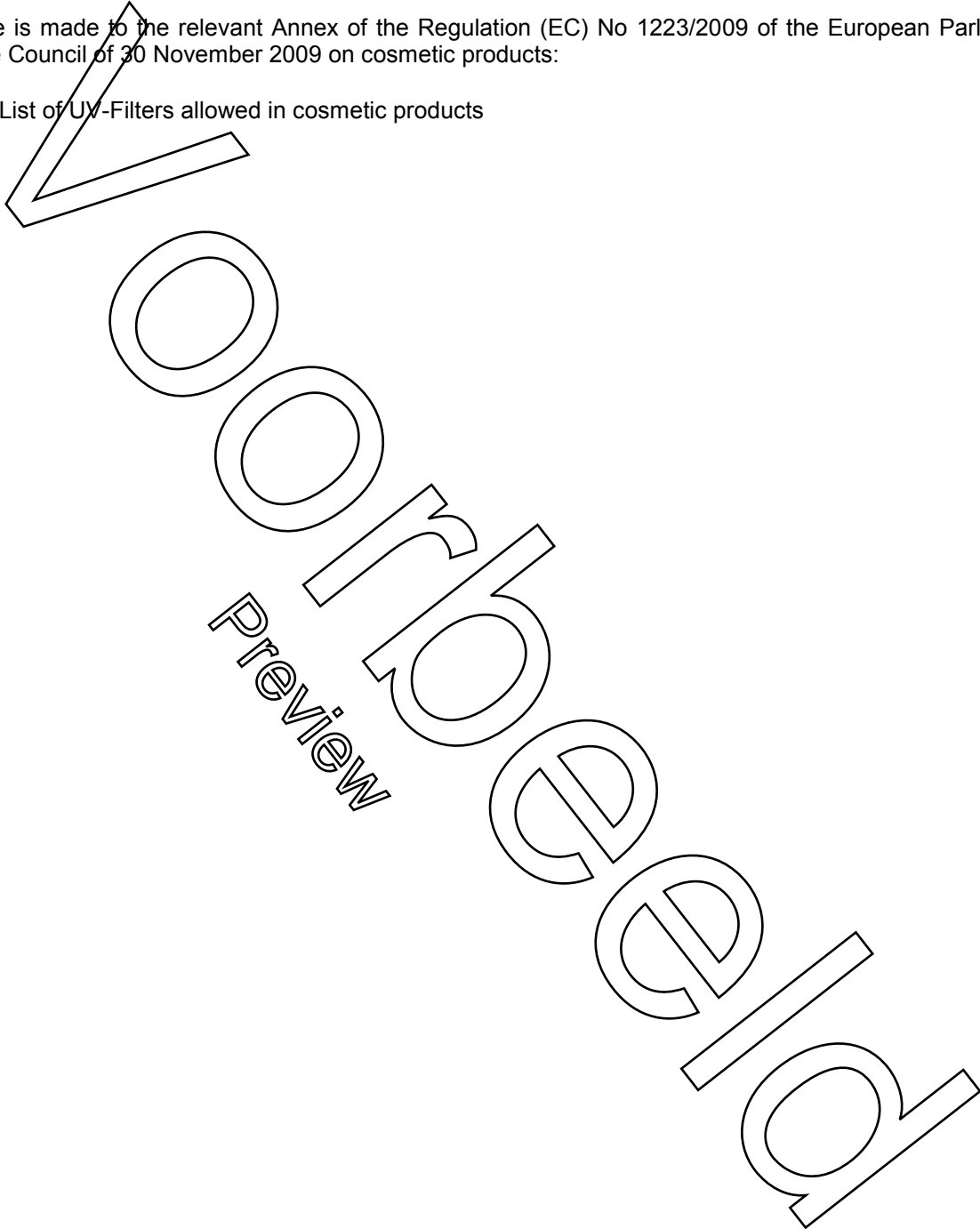
This document is currently submitted to the CEN Enquiry.



## Introduction

Reference is made to the relevant Annex of the Regulation (EC) No 1223/2009 of the European Parliament and of the Council of 30 November 2009 on cosmetic products:

Annex VI List of UV-Filters allowed in cosmetic products



## 1 Scope

This draft standard describes a multi-screening method using reversed-phase HPLC for the detection of UV filters listed in Annex VI of the Regulation (EC) No 1223/2009. The method is applicable for the quantitative determination of 10 UV filters which are mainly used in emulsion-based sun screen products and sun screen sprays particularly with regard to the maximum concentration listed in Annex VI.

Other analytical methods for the qualification and quantification of UV filters may be used if they lead to comparable results.

## 2 Terms and definitions

For the purposes of this document the following terms and definitions apply.

### 2.1

#### UV filters

organic chemical compounds that absorb ultraviolet light and inorganic substances that reflect, scatter and absorb UV light, they are used in sunscreen products to protect the skin against UV radiation

NOTE The UV filters and UV absorber of this method are only organic chemical compounds.

## 3 Principle

The UV filters are extracted with an acetone/methanol mixture. For the qualitative detection of the listed UV filters and the quantitative determination of the 10 validated UV-filter reversed phase HPLC with UV (DAD) detection is used. The method is also applicable for the quantification of the other listed UV filters after proper validation.

For the quantitative determination of samples containing the following UV-filters additional extraction methods and determinations are required

Terephthalylidene Dicamphor Sulfonic Acid (TDSA) and Disodium Phenyl Dibenzimidazole Tetrasulfonate (DPDT) are extracted additionally with methanolic-aqueous sodium hydroxide solution.

Methylene Bis-benzotriazolyl Tetramethylbutylphenol (MBBT) and Bis-Ethylhexyloxyphenol Methoxyphenyl Triazine (BEMT) are extracted additionally with a tetrahydrofuran/acetone mixture.

In the case of an unsatisfactory peak shape Butyl Methoxydibenzoylmethane (BMDM) is extracted additionally with an acetone/methanol/EDTA mixture.

The quantitative determination is made by means of RP-HPLC with UV (DAD). The UV-spectra are compared with the reference spectra in a database.

The concentration of each UV filter determined in accordance with this method is given in g/100 g.

## 4 Reagents

### 4.1 General

If not otherwise specified, analytical-grade chemicals shall be used; the water shall be distilled or of a corresponding purity. "Solution" shall be understood as an aqueous solution unless otherwise specified.

4.2 **Methanol (MeOH)**, HPLC grade

4.3 **Acetone**, HPLC grade

4.4 **Tetrahydrofuran (THF)**, HPLC grade

4.5 **Ammonia solution**, mass fraction  $\omega = 25$  g/100 g

4.6 **Sodium hydroxide solution**, molar concentration  $c = 1$  mol/l

4.7 **Ethylenediaminetetraacetic acid (EDTA) disodium salt dihydrate** ( $\text{Na}_2\text{EDTA} \times 2\text{H}_2\text{O}$ , CAS 6381-92-6, purity  $\geq 99$  %)

### 4.8 EDTA solution

Weigh 1,8 g of EDTA disodium salt dihydrate (4.7) into a 100 ml volumetric flask and fill up to the calibration mark with water.

4.9 **Ethanol**, HPLC grade

4.10 **Lauryl Trimethyl Ammonium Bromide** (LTAB, synonym: dodecyltrimethylammonium bromide, CAS 1119-94-4) if possible HPLC quality (purity  $\geq 98$  %)

4.11 **Ammonium bromide** (CAS 12124-97-9, purity  $\geq 99$  %)



## 4.12 Reference substances

Table 1 — Calibration solution polar (in methanol)

	EU <sup>a</sup>	Abbrev.	INCI <sup>b</sup> and other common names
4.12.1	A2	CBM	Camphor Benzalkonium Methosulfate, CAS 52793-97-2, e.g. Mexoryl SO, 29 % solution (on request from L'Oréal)
4.12.2	A6	PBSA	Phenylbenzimidazole Sulfonic Acid (2-phenylbenzimidazole-5-sulfonic acid), CAS 27503-81-7, e.g. Aldrich
4.12.3	A7	TDSA	Terephthalylidene Dicumyl Sulfonic Acid, CAS 90457-82-2, e.g. Mexoryl SX, Chimex; present as triethanolamine salt (molecular weight $m = 860$ g/Mol), free acid (molecular weight $m = 562$ g/Mol)
4.12.4	A22	B-4/5	Benzophenone-4/5 (2-hydroxy-4-methoxybenzophenone-5-sulfonic acid, Sulisobenzone), CAS 4065-45-6, e.g. Fluka
4.12.5	A24	DPDT	Disodium Phenyl Dibenzimidazole Tetrasulfonate, CAS 180898-37-7, e.g. Neo Heliopan AP, Symrise
4.12.6	A28	DHHB	Diethylamino Hydroxybenzoyl Hexyl Benzoate, CAS 302776-68-7, e.g. Uvinul A Plus, BASF

<sup>a</sup> EU = serial number in accordance with Annex VI of (EC) No 1223/2009.

<sup>b</sup> INCI = International Nomenclature of Cosmetic Ingredients.

Table 2 — Calibration solution medium polar (in methanol acetone (1:1))

	EU	Abbrev.	INCI and other common names
4.12.7	A4	B-3	Benzophenone-3 (oxybenzonom, 2-hydroxy-4-methoxy-benzophenone), CAS 131-57-7, e.g. Fluka
4.12.8	A10		Octocrylene (2-ethylhexyl-2-cyano-3,3-diphenylacrylate), CAS 6197-30-4, e.g. Aldrich
4.12.9	A12	EHMC	Ethylhexyl Methoxycinnamate (octylmethoxycinnamate), CAS 5466-77-3, e.g. Merck
4.12.10	A14	IMC	Isoamyl p-Methoxycinnamate, CAS 71617-10-2, e.g. Neo Heliopan E1000, Symrise
4.12.11	A18	MBC	4-Methylbenzylidene Camphor (3-(4-methylbenzylidene)-dl-camphor), CAS 36861-47-9, e.g. Merck
4.12.12	A19	3-BC	3-Benzylidene Camphor, CAS 15087-24-8, e.g. Unisol S-22, Induchem
4.12.13	A21	EHDP	Ethylhexyl Dimethyl PABA (2-ethylhexyl-4-dimethylaminobenzoate), CAS 21245-02-3, e.g. Aldrich

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