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Kosmetika – Analys av kosmetiska produkter – Screening av UV-filter i kosmetiska produkter och kvantitativ bestämning av 10 UV-filter med HPLC

Cosmetics – Analysis of cosmetic products – Screening for UV-filters in cosmetic products and quantitative determination of 10 UV-filters by HPLC

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EUROPEAN STANDARD

EN 16344

NORME EUROPÉENNE

EUROPÄISCHE NORM

July 2013

ICS 71.100.70

English Version

**Cosmetics - Analysis of cosmetic products - Screening for UV-
filters in cosmetic products and quantitative determination of 10
UV-filters by HPLC.**

Cosmétiques - Analyse des produits cosmétiques -
Détection des filtres UV dans les produits cosmétiques et
détermination quantitative de 10 filtres UV par CLHP

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

This European Standard was approved by CEN on 29 May 2013.

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

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Foreword

This document (EN 16344:2013) has been prepared by Technical Committee CEN/TC 392 “Cosmetics”, the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by January 2014, and conflicting national standards shall be withdrawn at the latest by January 2014.

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SS-EN 16344:2013 (E)

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 European Standard specifies a multi-screening method using reversed-phase HPLC for the detection of UV-filters listed in the cosmetic regulations. The method is applicable for the quantitative determination of 10 UV-filters, which are mainly used in emulsion-based cosmetic products and sunscreen sprays particularly with regard to the maximum concentration listed in the cosmetic regulation.

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

Note 1 to entry: The UV-filters and UV-absorber of this method are only organic chemical compounds and are used in sunscreen products to protect the skin against UV radiation.

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.

Quantitative determination of samples containing the following UV-filters require the use of additional extraction methods and determinations:

- Terephthalylidene Dicamphor Sulfonic Acid (TDSA) and Disodium Phenyl Dibenzimidazole Tetrasulfonate (DPDT) are additionally extracted with methanolic-aqueous sodium hydroxide solution.
- Methylene Bis-benzotriazolyl Tetramethylbutylphenol (MBBT) and Bis-Ethylhexyloxyphenol Methoxyphenyl Triazine (BEMT) are additionally extracted with a mixture of tetrahydrofuran/acetone.

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

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 reported in g/100 g.

This method has been tested in an inter-laboratory test on specific cosmetic matrix (q.v. Annex A). The user should verify the performance of the method in their laboratory for each different matrix and pay particular attention to the recommended quality control elements.

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4 Reagents

4.1 General

If not otherwise specified, analytical-grade chemicals shall be used. 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 $w = 25$ g/100 g.

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

4.7 Ethylenediaminetetraacetic acid (EDTA) disodium salt dihydrate ($\text{Na}_2\text{EDTA} \cdot 2\text{H}_2\text{O}$, CAS 6381-92-6, purity > 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 ≥ 98 %).

4.11 Ammonium bromide (CAS 12124-97-9, purity ≥ 99 %).

4.12 Reference substances

Table 1 — Polar UV-filters (calibration solution in methanol)

	EU ^a	Abbrev.	INCI ^b and other common names
4.12.1	A2	CBM	Camphor Benzalkonium Methosulfate, CAS 52793-97-2
4.12.2	A6	PBSA	Phenylbenzimidazole Sulfonic Acid (2-phenylbenzimidazole-5-sulfonic acid), CAS 27503-81-7
4.12.3	A7	TDSA	Terephthalylidene Dicapmor Sulfonic Acid, CAS 90457-82-2, present as triethanolamine salt (molecular weight <i>m</i> = 860 g/mol), free acid (molecular weight <i>m</i> = 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
4.12.5	A24	DPDT	Disodium Phenyl Dibenzimidazole Tetrasulfonate, CAS 180898-37-7
4.12.6	A28	DHNB	Diethylamino Hydroxybenzoyl Hexyl Benzoate, CAS 302776-68-7

^a EU = serial number in accordance with Annex VI of (EC) No 1223/2009.
^b INCI = International Nomenclature of Cosmetic Ingredients.

Table 2 — Medium polar UV-filters (calibration solution in methanol acetone (1:1))

	EU ^a	Abbrev.	INCI ^b and other common names
4.12.7	A4	B-3	Benzophenone-3 (oxybenzoniun, 2-hydroxy-4-methoxy-benzophenone), CAS 131-57-7
4.12.8	A10	OC	Octocrylene (2-ethylhexyl-2-cyano-3,3-diphenylacrylate), CAS 6197-30-4
4.12.9	A12	EHMC	Ethylhexyl Methoxycinnamate (octylmethoxycinnamate), CAS 5466-77-3
4.12.10	A14	IMC	Isoamyl p-Methoxycinnamate, CAS 71617-10-2
4.12.11	A18	MBC	4-Methylbenzylidene Camphor (3-(4-methylbenzylidene)-dl-camphor), CAS 36861-47-9
4.12.12	A19	3-BC	3-Benzylidene Camphor, CAS 15087-24-8
4.12.13	A21	EHDP	Ethylhexyl Dimethyl PABA (2-ethylhexyl-4-dimethylaminobenzoate), CAS 21245-02-3

^a EU = serial number in accordance with Annex VI of (EC) No 1223/2009.
^b INCI = International Nomenclature of Cosmetic Ingredients.

Table 3 — Non polar UV-filters (calibration solution in THF)

	EU ^a	Abbrev.	INCI ^b and other common names
4.12.14	A3	HMS	Homosalate (3,3,5-trimethylcyclohexylsalicylate), CAS 118-56-9
4.12.15	A8	BMDM	Butyl Methoxydibenzoylmethane (4-tert-butyl-4'-methoxydibenzoylmethane), CAS 70356-09-1
4.12.16	A15	EHT	Ethylhexyl Triazone (octyltriazone), CAS 88122-99-0
4.12.17	A16	DTS	Drometrizole Trisiloxane (2-benzotriazole-2-yl-methylphenol trisiloxane), CAS 155633-54-8
4.12.18	A17	DEBT	Diethylhexyl Butamido Triazone, CAS 154702-15-5
4.12.19	A20	EHS	Ethylhexyl Salicylate (2-ethylhexylsalicylate), CAS 118-60-5
4.12.20	A23	MBBT	Methylene Bis-Benzotriazol Tetramethylbutylphenol, CAS 103597-45-1
4.12.21	A25	BEMT	Bis-Ethylhexyloxyphenol Methoxyphenyl Triazine (anisotriazine), CAS 187393-00-6

^a EU = serial number in accordance with Annex VI of (EC) No 1223/2009.
^b INCI = International Nomenclature of Cosmetic Ingredients.