
Kozmetika - Analiza kozmetičnih izdelkov - Detekcija UV-filtrov v kozmetičnih izdelkih in kvantitativno določevanje 10 UV-filtrov s tekočinsko kromatografijo visoke ločljivosti (HPLC)

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

Kosmetische Mittel - Untersuchung von kosmetischen Mitteln - Screening und quantitative Bestimmung von 10 UV-Filtern in Sonnenschutzmitteln, HPLC-Verfahren
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Cosmétiques - Analyse des produits cosmétiques - Détection des filtres UV dans les produits cosmétiques et détermination quantitative de 10 filtres UV par CLHP

Ta slovenski standard je istoveten z: EN 16344:2013

ICS:

71.100.70 Kozmetika. Toaletni Cosmetics. Toiletries
 pripomočki

SIST EN 16344:2013

en,fr,de

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EUROPEAN STANDARD

EN 16344

NORME EUROPÉENNE

EUROPÄISCHE NORM

July 2013

ICS 71.100.70

English Version

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Détection des filtres UV dans les produits cosmétiques et
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UV-Filtern in Sonnenschutzmitteln, HPLC-Verfahren

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of an identical text or by endorsement, at the latest by January 2014, and conflicting national standards shall be withdrawn at the latest by January 2014.

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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.

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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

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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.

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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.

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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 %).

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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 Dicamphor Sulfonic Acid, CAS 90457-82-2, 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
4.12.5	A24	DPDT	Disodium Phenyl Dibenzimidazole Tetrasulfonate, CAS 180898-37-7
4.12.6	A28	DHHB	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.

Table 4 — Other UV-filter or UV absorber (not listed in Annex VI of the (EC) No 1223/2009)

		Abbrev.	INCI ^b and other common names
4.12.22		MA	Menthyl Anthranilate, CAS 134-09-8
4.12.23		PABA	PABA (4-aminobenzoic acid), CAS 150-13-0
4.12.24		B-1	Benzophenone-1 (2,4-dihydroxybenzophenone), CAS 131-56-6
4.12.25		B-2	Benzophenone-2 (2,2',4,4'-tetrahydroxybenzophenone), CAS 131-55-5
4.12.26		DPLT	Dimethyl-PABAamidopropyl Laurdimonium Tosylate, CAS 156679-41-3
4.12.27		B-6	Benzophenone-6 (2,2'-dihydroxy-4,4'-dimethoxybenzophenone), CAS 131-54-4
4.12.28		B-8	Benzophenone-8 (2,2'-dihydroxy-4-methoxybenzophenone, dioxymethoxybenzophenone), CAS 131-53-3
4.12.29		B-9	Benzophenone-9 (disodium 3,3'-carbonylbis[4-hydroxy-6-methoxybenzenesulphonate]), CAS 76656-36-5
4.12.30		B-10	Benzophenone-10 (2-hydroxy-4-methoxy-4'-methylbenzophenone), CAS 1641-17-4
4.12.31		SA	Sodium Salicylate, CAS 54-21-7

^b INCI = International Nomenclature of Cosmetic Ingredients.

NOTE The following UV-filters listed in Annex VI of the Regulation (EC) No 1223/2009 are not part of this method:

- A 9: 3-(4'-sulfo)-benzylidene-bornan-2-one, CAS 56039-58-8
- A 11: polymer of N-(2(and 4)-(2-oxoborn-3-ylidenemethyl)benzyl)acrylamid, CAS 113783-61-2 (the substances are no longer commercially available)
- A 13: ethoxylated ethyl-4-aminobenzoate, CAS 116242-27-4 (can be determined only qualitatively)
- A 26: dimethicodiethylbenzalmalonate, CAS 207574-74-1 (cannot be determined using this standard)

4.13 Extraction solution

4.13.1 Acetone/methanol mixture (for the preparation of the reference solutions and for extraction).

Mix 500 ml of acetone (4.3) and 500 ml of methanol (4.2) in a 1 000 ml conical flask.

4.13.2 Acetone/tetrahydrofuran mixture (for extraction of non-polar UV-filters).

Mix 500 ml of acetone (4.3) and 500 ml of tetrahydrofuran (4.4) in a 1 000 ml conical flask.

4.13.3 Methanolic-aqueous sodium hydroxide solution (for extraction of polar UV-filters).

Mix 800 ml of methanol (4.2) and 200 ml of water and 10 ml of sodium hydroxide solution (4.6) in a 1 000 ml conical flask.

4.13.4 Acetone/methanol/EDTA mixture (for extraction of BMDM-containing samples with unsatisfactory peak shape).

Add 1,0 ml of EDTA solution (4.8) to 200 ml of acetone/methanol mixture (4.13.1) in a 200 ml conical flask. As the solution is supersaturated with regard to EDTA it shall be used on the same day.

4.14 Reference solutions

4.14.1 Stock solutions

The stock solutions are prepared in accordance with Table 5, Table 6 and Table 7. For each substance, weigh the initial weights with an accuracy of 0,1 mg to the mentioned volume. One stock solution is prepared per substance. For the initial weight, the purity of the substances shall be taken into account and if applicable be converted to an initial weight of 4 mg/ml of "active substance" in each case. The stock solutions can be stored for at least one month in a refrigerator between 2 °C and 8 °C in the absence of light.

Table 5 — Scheme for preparing the stock solutions of polar UV-filters

Polar UV-filter	Initial weight	Volume	Solvent
CBM (29 %) (4.12.1)	140 mg	10 ml	Methanol (4.2) (Initial weight: 140 mg, as a 29 % standard solution is used; corresponds to 40 mg of "active substance")
PBSA (4.12.2)	40 mg	10 ml	Add 2 ml of methanol (4.2) and 3 drops of ammonia (4.5). Fill up to the calibration mark with water.
TDSA (4.12.3)	60 mg	10 ml	Add 2 ml of methanol (4.2) and 3 drops of ammonia (4.5). Fill up to the calibration mark with water. (Initial weight: 60 mg because of triethanolamine salt; corresponds to 40 mg of free acid, see limit value)
B-4/5 (4.12.4)	40 mg	10 ml	Methanol (4.2)
DPDT (4.12.5)	40 mg	10 ml	Add 2 ml of methanol (4.2) and 3 drops of ammonia (4.5). Fill up to the calibration mark with water.
DHHB (4.12.6)	40 mg	10 ml	Methanol (4.2)

Table 6 — Scheme for preparing the stock solutions of medium polar UV-filters

Medium-polar UV-filter	Initial weight	Volume	Solvent
B-3 (4.12.7)	40 mg	10 ml	Acetone/methanol mixture (4.13.1)
OC (4.12.8)	40 mg	10 ml	Acetone/methanol mixture (4.13.1)
EHMC (4.12.9)	40 mg	10 ml	Acetone/methanol mixture (4.13.1)
MBC (4.12.11)	40 mg	10 ml	Acetone/methanol mixture (4.13.1)
IMC (4.12.10)	40 mg	10 ml	Acetone/methanol mixture (4.13.1)
3-BC (4.12.12)	40 mg	10 ml	Acetone/methanol mixture (4.13.1)
EHDP (4.12.13)	40 mg	10 ml	Acetone/methanol mixture (4.13.1)