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Kosmetik – Analytiska metoder – LC/UV -metod för identifiering och kvantitativ bestämning i kosmetiska produkter av de 22 organiska UV-filtren som används i EU

Cosmetics – Analytical methods – LC/UV method for the identification and quantitative determination in cosmetic products of the 22 organic UV filters in use in the EU

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EUROPEAN STANDARD

EN 17156

NORME EUROPÉENNE

EUROPÄISCHE NORM

December 2018

ICS 71.100.70

English Version

Cosmetics - Analytical methods - LC/UV method for the identification and quantitative determination in cosmetic products of the 22 organic UV filters in use in the EU

Cosmétiques - Méthodes analytiques - Procédé CL/UV pour l'identification et la détermination quantitative des 22 filtres UV organiques utilisés dans les produits cosmétiques au sein de l'UE

Kosmetische Mittel - Untersuchungsverfahren - LC/UV Verfahren für die Identifizierung und qualitative Bestimmung von den 22 in der EU verwendeten organischen UV-Filtern in kosmetischen Produkten

This European Standard was approved by CEN on 5 November 2018.

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (EN 17156:2018) 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of an identical text or by endorsement, at the latest by June 2019 and conflicting national standards shall be withdrawn at the latest by June 2019.

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Introduction

In order to protect human skin from the deleterious UV radiation of sunlight, the so-called UV filters have been used as active ingredients in the formulation of sunscreen cosmetic products. These active compounds are of organic or inorganic nature, and have the capacity to absorb and/or reflect, respectively, this UV radiation. Nowadays, they are not only added to those cosmetics intended specifically for sun protection but also in all type of daily products such as moisturizers, after shave products, shampoos, anti-aging creams, make-up products, etc.

The compounds that can be used as UV filters in cosmetics and their maximum allowed concentrations are regulated in order to ensure user's safety. Currently, the European Union (EU) 1223/2009 Regulation permits the use of 27 compounds as UV filters [1], the names of which are listed in Table 1. Among these 27 UV filters, titanium dioxide and zinc oxide are of inorganic nature, and among the remaining 25, 7 are highly polar and can be grouped in the 'water-soluble' group, whereas the other 18 are low polar and can be grouped in the 'fat-soluble' group. All these compounds are included in the formulations of the different cosmetic products consumed in the EU framework, with the only exceptions of BCSA and PBC which are not being currently used.

Besides, errors during the manufacturing process of cosmetics may cause a lower concentration in the final product than that formulated. This might affect the efficacy of the product since the real Sun Protection Factor could be lower than that labelled.

Therefore, reliable and practical analytical methods are needed in order to ensure compliance with the EU Regulation and thus protect user's safety, but also to ensure product's efficacy.

In this sense, with the aim of implementing a broad-spectrum analytical method to improve and facilitate the quality control of the cosmetic industry, this European Standard presents an analytical method for the quantification of 22 organic UV filters. They constitute all the organic UV filters allowed and in use in the EU when this standard was validated. Note that TBT is not included since it was later approved. The presented method, besides good analytical characteristics, is simple, low cost, rapid and both user- and environmentally-friendly. The method is based on different analytical methods previously published by the Research Group on Quality Control of Cosmetic Products of the University of Valencia [2], [3].

Table 1 — List of the UV filters permitted in cosmetic products under the EU Regulation

| EU Reference number^a | Name of Common Ingredients Glossary^b | Acronym^c |
|--|--|----------------------------|
| 2 | Camphor Benzalkonium Methosulfate | CBM |
| 3 | Homosalate | HMS |
| 4 | Benzophenone-3 | BZ3 |
| 6 | Phenylbenzimidazole Sulfonic Acid | PBSA |
| 7 | Terephthalylidene Dicamphor Sulfonic Acid | TDSA |
| 8 | Butyl Methoxydibenzoylmethane | BMDM |
| 9 | Benzylidene Camphor Sulfonic Acid | BCSA |
| 10 | Octocrylene | OC |
| 11 | Polyacrylamidomethyl Benzylidene Camphor | PBC |
| 12 | Ethylhexyl Methoxycinnamate | EHMC |
| 13 | PEG-25 PABA | P25 |
| 14 | Isoamyl p-Methoxycinnamate | IMC |
| 15 | Ethylhexyl Triazone | EHT |
| 16 | Drometrizole Trisiloxane | DTS |
| 17 | Diethylhexyl Butamido Triazone | DEBT |
| 18 | 4-Methylbenzylidene Camphor | MBC |
| 20 | Ethylhexyl Salicylate | EHS |
| 21 | Ethylhexyl Dimethyl PABA | EHDP |
| 22 | Benzophenone-4 | BZ4 |
| 23 | Methylene Bis-Benzotriazolyl Tetramethylbutylphenol | MBBT |
| 24 | Disodium Phenyl Dibenzimidazole Tetrasulfonate | PDTA |
| 25 | Bis-Ethylhexyloxyphenol Methoxyphenyl Triazine | BEMT |
| 26 | Polysilicone-15 | P15 |
| 27 | Titanium Dioxide | TiO ₂ |
| 28 | Diethylamino Hydroxybenzoyl Hexyl Benzoate | DHHB |
| 29 | Tris-biphenyl triazine | TBT |
| 30 | Zinc Oxide | ZnO |

^a Order number given according to the EU 1223/2009 Regulation.

^b According to the Annex VI of the European Union (EU) 1223/2009 Regulation.

^c Acronyms used in this standard.

SS-EN 17156:2018 (E)

1 Scope

This document specifies an analytical method, based on liquid-chromatography (LC) with ultraviolet/visible spectrometry (UV/Vis) detection for the detection and quantitative determination of 22 organic UV filters in use in the EU framework. This method has been validated for emulsion-based cosmetic products, lip-balms, lotions and waters.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

4 Principles

The cosmetic sample is weighed and solved in ethanol. For difficult-to-solve samples, the use of ultrasound or vortex can help. After that, depending on the UV filters to be determined, aliquots are taken and diluted with a mixture of ethanol:acetate buffer, ethanol or tetrahydrofuran for the determination of the water-soluble group, the fat-soluble group or P15 (which is a polymer), respectively.

Regarding the water-soluble group, the 6 UV filters are measured by employing a reversed-phase column and a mobile phase of ethanol and acetate buffer. Regarding the fat-soluble group, the 15 UV filters are determined by employing a reversed-phase column and a mobile phase of ethanol and aqueous formic acid containing hydroxypropyl-beta-cyclodextrin as mobile phase modifier. Finally, for P15 determination, a size-exclusion column and tetrahydrofuran as mobile phase are used.

5 Reagents

If not otherwise specified, analytical-grade chemicals shall be used.

- 5.1 **Ethanol (EtOH)**, LC-grade.
- 5.2 **Tetrahydrofuran (THF)**, LC-grade.
- 5.3 **Deionized water**, 18,2 M Ω ·cm resistivity.
- 5.4 **2-Hydroxypropyl- β -cyclodextrin (HP- β -CD)**, 1 309 g/mol molecular weight.
- 5.5 **Formic acid**, 98 %, d = 1,22 g/ml.
- 5.6 **Glacial acetic acid**, > 99 %, d = 1,05 g/ml.
- 5.7 **Sodium hydroxide**, pellets.
- 5.8 **Sodium hydroxide**, 10 % (w/w) aqueous solution.

Weigh 10 g of sodium hydroxide (5.7) and dissolve in 100 ml of deionized water (5.3).

5.9 Acetate buffer, 1 % (v/v) solution, pH = 4,75.

Take 10 ml of glacial acetic acid (5.6) and dilute with approximately 900 ml of deionized water (5.3). Adjust with the aid of pH-meter up to pH 4,75 by adding sodium hydroxide solution (5.8). Complete up to 1 l with deionized water (5.3).

5.10 Formic acid, 1 % (v/v) solution, containing 20 mmol HP- β -CD.

Weigh 26,2 g of HP- β -CD (5.4). Add 10 ml of formic acid (5.5), and complete up to 1 l with deionized water (5.3).

5.11 UV filter standards, see Table 2.**Table 2 — UV filter standards**

| Type | UV filter | CAS |
|---|---|-------------|
| Water-soluble UV filters | Disodium Phenyl Dibenzenimidazole Tetrasulfonate (PDTA) | 180898-37-7 |
| | Phenylbenzimidazole Sulfonic Acid (PBSA) | 27503-81-7 |
| | Terephthalylidene Dicamphor Sulfonic Acid (TDSA) (triethanolamine salt) | 90457-82-2 |
| | Benzophenone-4 (BZ4) | 4065-45-6 |
| | Camphor Benzalkonium Methosulfate (CBM) (29 % aqueous solution) | 52793-97-2 |
| | PEG-25 PABA (P25) | 116242-27-4 |
| Fat-soluble UV filters | Benzophenone-3 (BZ3) | 131-57-7 |
| | Isoamyl p-Methoxycinnamate (IMC) | 71617-10-2 |
| | 4-Methylbenzylidene Camphor (MBC) | 36861-47-9 |
| | Diethylamino Hydroxybenzoyl Hexyl Benzoate (DHHB) | 302776-68-7 |
| | Octocrylene (OC) | 6197-30-4 |
| | Ethylhexyl Dimethyl PABA (EHDP) | 21245-02-3 |
| | Butyl Methoxydibenzoylmethane (BMDM) | 70356-09-1 |
| | Ethylhexyl Methoxycinnamate (EHMC) | 5466-77-3 |
| | Ethylhexyl Salicylate (EHS) | 118-60-5 |
| | Homosalate (HMS) | 118-56-9 |
| | Diethylhexyl Butamido Triazone (DEBT) | 154702-15-5 |
| | Ethylhexyl Triazone (EHT) | 88122-99-0 |
| | Drometrizole Trisiloxane (DTS) | 155633-54-8 |
| | Methylene Bis-Benzotriazolyl Tetramethylbutylphenol (MBBT) | 103597-45-1 |
| Bis-Ethylhexyloxyphenol Methoxyphenyl Triazine (BEMT) | 187393-00-6 | |
| Polymeric UV filter | Polysilicone-15 (P15) | 207574-74-1 |