

Cosmetics - Analytical methods - HPLC/UV method for the identification and assay of hydroquinone, ethers of hydroquinone and corticosteroids in skin whitening cosmetic products

EESTI STANDARDI EESSÕNA

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English Version

**Cosmetics - Analytical methods - HPLC/UV method for the
identification and assay of hydroquinone, ethers of
hydroquinone and corticosteroids in skin whitening
cosmetic products**

Cosmétiques - Méthodes analytiques - Méthode de
CLHP couplée à la détection UV pour l'identification et
l'analyse de l'hydroquinone, de ses éthers et des
corticostéroïdes dans les produits cosmétiques
éclaircissants de la peau

Kosmetische Mittel - Untersuchungsverfahren -
HPLC/UV Verfahren für die Identifizierung und
Bestimmung von Hydrochinon, Hydrochinonethern
und Kortikosteroiden in hautaufhellenden
kosmetischen Mitteln

This European Standard was approved by CEN on 19 June 2017.

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European foreword

This document (EN 16956:2017) 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of an identical text or by endorsement, at the latest by March 2018, and conflicting national standards shall be withdrawn at the latest by March 2018.

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.

The existing peer review validation data for hydroquinone are preliminary and will be supplemented by inter-laboratory test data if available.

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Introduction

Hydroquinone is not allowed for use in cosmetic products for skin whitening and depigmentation of dermal spots or imperfections. Due to its cytotoxic effects its use has been regulated. Hydroquinone and 3 of its ethers (hydroquinone monomethylether (MME), hydroquinone monoethylether (MEE) and hydroquinone monobenzylether (MBE)) are regulated by the cosmetic regulation 1223/2009. Nowadays the use of these substances is prohibited in skin whitening cosmetic products.

Depigmentation is a side effect of topical steroids, in this way corticosteroids might be used as compounds in products illegally sold as cosmetics. Corticosteroids most commonly found in these products are clobetasol propionate, fluocinonide, betamethasone dipropionate, and fluocinolone acetonide (see Figure 1). Corticosteroids are listed in Regulation 1223/2009 Annex II "List of substances prohibited in cosmetic products" (reference number 300), and their use is also prohibited in cosmetic products.

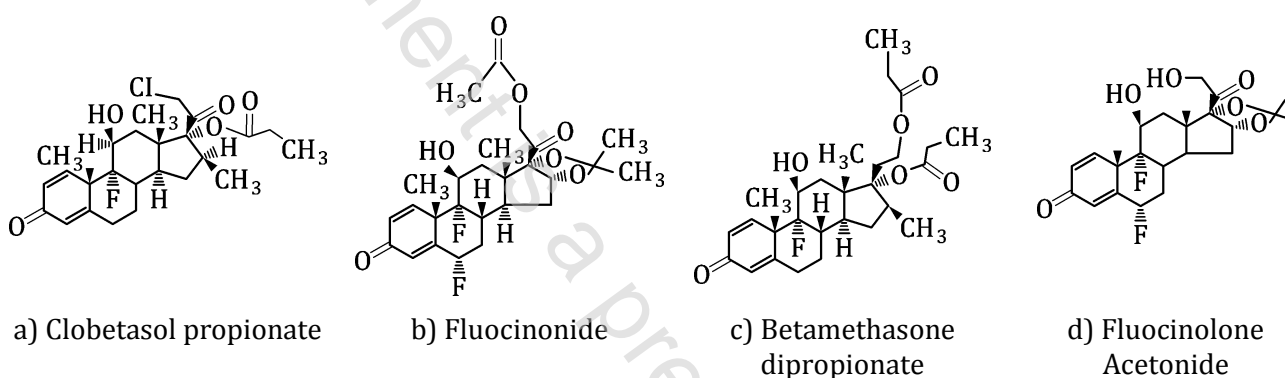


Figure 1 — Corticosteroids most commonly found in illegal cosmetics

All these substances work on the same principle as hydroquinone which mainly consists of inhibition of melanin synthesis.

The cosmetic directive 95/32/EC [2] gives an analytical method for the assay of hydroquinone and 3 of its ethers (hydroquinone monomethylether (MME), hydroquinone monoethylether (MEE) and hydroquinone monobenzylether (MBE)) in cosmetic products for lightening the skin. In order to update and extend this official method to the identification and assay of corticosteroids in cosmetic products, this standard describes an HPLC/UV method for the identification and assay of hydroquinone, ethers of hydroquinone and corticosteroids in cosmetic products.

1 Scope

This European Standard specifies a HPLC/UV method for the identification and quantification of hydroquinone, 3 ethers of hydroquinone and 4 corticosteroids most frequently found in illegally sold skin whitening cosmetic products: clobetasol propionate, betamethasone dipropionate, fluocinonide and fluocinolone acetonide.

This standard also gives HPLC/UV methods for the identification of 38 corticosteroids that may be found in skin whitening cosmetic products (see Annex D).

This standard is not dedicated to artificial nail products or soaps.

2 Principle

The sample is extracted by a mixture of water/methanol and gently warmed in order to extract compounds present in the product. The obtained mixture is filtered. The quantitation of present compounds in solution is made by reversed phase HPLC with DAD (Diode Array Detector) detection.

3 Reagents

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.

3.1 Methanol, HPLC grade.

3.2 Water, HPLC grade.

3.3 Extraction solution, methanol/water (1/1).

Mix 500 ml of methanol (3.1) and 500 ml of water (3.2) in a 1 000 ml conical flask.

3.4 Compounds considered, see Table 1.

Table 1 — Compounds considered

Compound	CAS	Manufacturer ^a	Purity %	Used method - clause
Alclometasone dipropionate (ACD)	66734-13-2	USP	99,2	Annex D
Amcinonide (AMC)	51022-69-6	Sigma	97,9	Annex D
Beclomethasone dipropionate (BCD)	5534-09-8	Sigma	99,0	Annex D
Betamethasone acetate (BMA)	987-24-6	Sigma	98,6	Annex D
Betamethasone (BM)	378-44-9	Sigma	98,4	Annex D
Betamethasone dipropionate (BMD)	5593-20-4	Sigma	98,6	2/ Annex D
Betamethasone valerate (BMV)	2152-44-5	Sigma	98,1	Annex D
Budesonide (BUD)	51333-22-3	Ph. Eur.	99,7	Annex D
Clobetasol propionate (CP)	25122-46-7	Sigma	98,8	2/ Annex D
Clocortolone pivalate (CLP)	34097-16-0	USP	98,9	Annex D
Cortisone (CS)	53-06-5	Sigma	98,3	Annex D
Desonide (DSN)	638-94-8	Cil	98,0	Annex D