

High-performance liquid chromatographic determination of α -tocopheryl nicotinate in cosmetic preparations

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ABSTRACT

α -Tocopheryl nicotinate (α -TN) accelerates blood circulation and stimulates hair follicle cells, hence it is an active ingredient in a broad range of cosmetic products. A reversed-phase high-performance liquid chromatographic method was developed to determine α -TN in cosmetic preparations with α -tocopheryl acetate as internal standard. The method was found to be rapid, precise and specific.

INTRODUCTION

Various derivatives of tocopherol are employed in drugs as peripheral vasodilators [1]. In particular, nicotinic acid esters (methyl and benzyl) are mainly used in cosmetic preparations as circulation accelerators. α -Tocopheryl nicotinate (α -TN) combines the effects of its constituents, vitamin E and nicotinic acid [2], without displaying the adverse side-effects shown by other nicotinate derivatives, *i.e.*, erythema and heat sensation. α -TN accelerates blood circulation and stimulates hair follicle cells, hence it is an active ingredient in a broad range of cosmetic products [3-12] such as skin conditioners (lotions and creams) and hair growth-promoters (lotions and shampoos).

Some reversed-phase high-performance liquid chromatographic (HPLC) methods for the separation of tocopherol derivatives have been reported [13-16]. Only one study considered the determination of α -TN in plasma [16] but it was not found suitable for monitoring this compound in cosmetics.

This paper describes a rapid and specific method for the determination of α -TN in cosmetic preparations.

EXPERIMENTAL

Materials

α -Tocopheryl nicotinate (α -TN) and the internal

standard, α -tocopheryl acetate (α -TA), were purchased from Sigma (St. Louis, MO, USA) and Carlo Erba (Milan, Italy), respectively. Span 60 was obtained from Fluka (Buchs, Switzerland), Tween 61 from Auschem (Milan, Italy), stearic acid, stearyl alcohol, Tween 80, propylparaben, propylene glycol and methylparaben from Aldrich-Chemie (Steinheim, Germany), squalane, ethoxylated hydrogenated lanolin, alkylimidoabetaïne, cocamide DEA and coconut oil from Esperis (Milan, Italy), sodium lauryl sulphate from Janssen (Beerse, Belgium) and mineral oil, triethanolamine and ethanol from Carlo Erba. All solvents used were of HPLC grade.

Equipment

Determinations were performed on an HP 1090M liquid chromatograph (Hewlett-Packard, Palo Alto, CA, USA) equipped with a sample valve (Rheodyne Model 7410) with a 10- μ l loop, connected to an HP 1090M UV-VIS diode-array detector controlled by an HP 9000 Model 310 workstation.

Analytical conditions

The column was ODS Hypersil C₁₈ (100 \times 4.6 mm I.D.) (5- μ m spherical particles) from Hewlett-Packard (Cernusco sul Naviglio, Milan, Italy).

Isocratic elution was carried out with the mobile phase methanol-water-2-propanol (97:2:1, v/v/v) at a flow-rate of 1 ml/min. The analytes were monitored at 210 nm (bandwidth 4 nm).

Standard solutions

A 1 mg/ml standard solution of α -TN in 2-propanol was prepared and diluted to obtain concentrations ranging from 10 to 100 μ g/ml. A 1 mg/ml stock standard solution of α -TA internal standard was prepared in 2-propanol. The solutions were kept in brown bottles, refrigerated at 4°C.

Cosmetic preparations

Four laboratory-made products were prepared containing the common concentrations of α -TN and excipients and formulations as reported [12].

A skin cream was prepared consisting of 3 g of Span 60, 3 g of Tween 61, 8 g of coconut oil, 0.5 g of α -TN, 6 g of stearic acid, 5 g of stearyl alcohol, 7 g of squalane, 0.1 g of propylparaben, 8 g of ethoxylated hydrogenated lanolin, 5 g of propylene glycol, 0.2 g of methylparaben and deionized water to 100 g.

A skin milk was prepared consisting of 10 g of mineral oil, 1 g of stearyl alcohol, 3 g of stearic acid, 0.5 g of α -TN, 1.8 g of triethanolamine, 0.2 g of methylparaben and deionized water to 100 g.

A hair lotion was prepared consisting of 0.3 g of α -TN, 40 g of ethanol, 2 g of Tween 80, 5 g of propylene glycol and deionized water to 100 g.

A shampoo was prepared consisting of 0.5 g of α -TN, 4 g of sodium lauryl sulphate, 25 g of alkylimidobetaine (30%), 2 g of cocamide DEA, 0.2 g of methylparaben and deionized water to 100 g.

Sample preparation

A 0.5-g and a 1.0-g sample of cosmetic product [for formulations containing 0.5% and 0.3% (w/w) of α -TN, respectively] were accurately weighed into a 100-ml volumetric flask, 2.5 ml of internal stan-

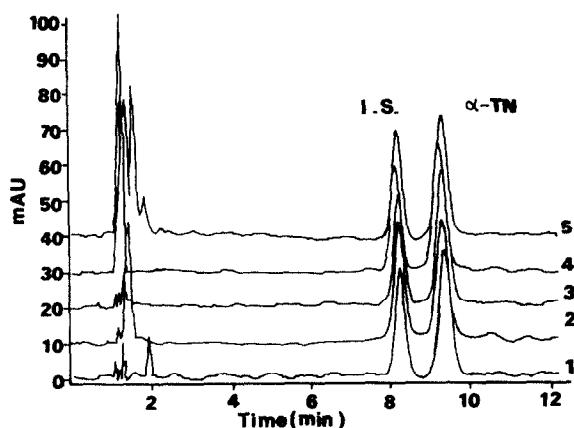


Fig. 1. Chromatograms of α -TN in different products: 1 = standard solution; 2 = skin milk; 3 = lotion; 4 = skin cream; 5 = shampoo. I.S. = Internal standard (α -TA).

dard stock solution and about 30 ml of 2-propanol were added and the mixture was stirred until complete solution was effected. The solutions were diluted to a final volume of 100 ml with 2-propanol and filtered over a FHP 0.5- μ m filter (Millipore) prior to injection.

RESULTS AND CONCLUSIONS

The chromatographic conditions described above were selected to obtain a good resolution of α -TN and peaks from excipients and to optimize the peak shape.

Typical chromatograms of α -TN standard and α -TN extracted from cosmetic samples are shown in Fig. 1. No interferences at the location of either the α -TN or α -TA peaks occurred.

TABLE I
DETERMINATION OF α -TN IN COSMETIC PREPARATIONS

Product	Amount added (%, w/w)	Average found ^a (%, w/w)	Mean recovery \pm S.D. (%)	R.S.D. ^b (%)
Skin cream	0.50	0.5091	101.62 \pm 0.9555	0.94
Skin milk	0.50	0.5074	101.48 \pm 0.7000	0.69
Lotion	0.30	0.3069	102.29 \pm 1.3858	1.35
Shampoo	0.50	0.5078	101.56 \pm 0.6581	0.64

^a Mean values for five samples.

^b Relative standard deviation.

A calibration graph was constructed by plotting the peak-height ratio (α -TN to internal standard) versus the concentration of α -TN. The response was linear in the range investigated (10–100 $\mu\text{g/ml}$, $n = 6$, $r = 0.999$, $y = 0.932x + 0.0503$). The equation describing the graph was selected to correspond to the common concentrations in cosmetic products.

For each laboratory-made preparation five samples were analysed in triplicate and the results are summarized in Table I.

The method was found to be rapid, precise and specific for application to the determination of α -TN in cosmetic preparations.

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