

N-NITROSODIETHANOLAMIN (NDELA) IN COSMETICS



Cosmetics, particularly mascara and skin care products, show partly very high concentration of the carcinogenic compound N-Nitrosodiethanolamine (NDELA).

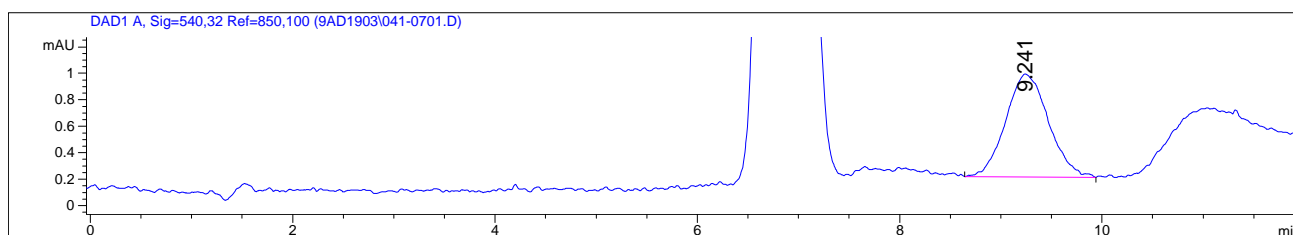
There are several methods for analysis available, e.g. GC-TEA, HPLC-MS/MS or HPLC with photolysis and Post-Column Derivatization. Mainly the last method is characterized by a high specificity, minimize the risk of measuring artefacts and allows an accurate quantification. Therefore is this method proposed by the Technical Committee for Cosmetics of the International Organization for Standardization. The procedure is described under the description ISO/WD 10130.

Description of Method

First of all NDELA will be separated from the cosmetic product and cleaned up using SPE- or Dichloromethane. After that, NDELA will be chromatographically separated from the matrix on a RP C18-HPLC column. The N-nitroso-bond is photochemically cracked at 254 nm using UV-Derivatization Module UVE™. Afterwards the becoming Nitrite moved to a Griess-reaction with a single Post Column Derivatization-System PINNACLE PCX to a strongly colored azo dye which can be detected at 540 nm.

Chromatogram

Calibration Dilution: 17,5 ng/mL NDELA



www.md-scientific.dk
Denmark

APPLICATION NOTE



HPLC Conditions and Derivatization Parameters

HPLC Conditions of the CVUA KA	
Operation Mode	Isocratic
Eluant	0,02M sodium acetate in water (pH 6,8)
Degassing	Helium degassed
HPLC Column	Spherisorb ODS-2, 4,6 x 150 mm with Precolumn 4,6 x 20 mm; C18,5 µm
Column Oven	50 °C
Flow Rate	0,5 mL/min
Post-Column Derivatization	
UVE™	Photochemical Reactor
Pinnacle PCX	Single Pump
Reactor Volume	1,0 mL
Reactor Temperature	65 °C
Reagent	Dissolve 0,25 g N-(1-naphthyl) ethylenediamine dihydrochloride in water and make up to 250 mL in a volumetric flask. Dissolve 4,0 g sulfanilamide in 250 mL of a 5% (w/v) aqueous solution of 85% orthophosphoric acid. Mix the reagents together in an amber glass bottle and keep the mixture away from the light.
Reagent Flow	0.5 mL/min
Detection	
Detection Type	UV/VIS detection
UV/VIS	540 nm, 32 with reference 850, 100 nm
Flow cell	Analytic; pressure stable up to 7 bar

Literature

A method for the determination of *N*-Nitrosodiethanolamine (NDELA) in Personal Care Products – Collaboratively evaluated by the CTPA Nitrosamines Working Group

Chris Flower¹, Stephen Carter², Andy Earls², Richard Fowler³, Stewart Hewlins⁴, Sam Lalljie⁵, Mark Lefebvre⁶, Jacqueline Mavro⁶, David Small⁷, and Nathalie Volpe⁸

¹Cosmetic Toiletry and Perfumery Association, Josaron House, 5-7 John Princes Street, London, W1G 0JN, U.K. Tel: (+44) 20 7491 8891, Fax: (+44) 20 7493 8061, E-mail: info@ctpa.org.uk

²Consumer Safety Department, LGC Limited, Queens Road, Teddington, Middlesex, TW11 0LY, U.K

³Quality Department, Boots Manufacturing, Nottingham, NG90 2PR, U.K

⁴Procter and Gamble Technical Centre, U.K Limited, Rusham Park, Whitehall Lane, Egham, TW20 9NW, UK

⁵Safety and Environmental Assurance Centre, Unilever Colworth, Sharnbrook, Bedford, MK44 1LQ, U.K

⁶Advanced Research - Analytical Chemistry- Physical & Chemical Sciences, L'Oreal Recherche, Avenue Eugène Schueller BP22 –F-93601 Aulnay sous Bois, France

⁷STIEFEL Laboratories, High Wycombe, Buckinghamshire, HP 100AU, U.K

⁸Christian Dior, Technical R&D, Laboratoire Chimie Fine, St Jean de Braye Cedex, 45804, France

1. John A. Casanova, Lois K. Gross, Sarah E. McMullen and Frank Schenck. (2006) J.AOAC Int. Vol. 89, No. 2, 447 – 451.
2. Use of Griess Reagents Containing Vanadium(III) for the Post-Column Derivatization and Simultaneous Determination of Nitrite and Nitrite in Baby Food, John A. Casanova, Lois K. Gross, Sarah E. McMullen and Frank Schenck, Food and Drug Administration, 60 8th Street, Atlanta, GA 30309.

Order Information

Order Number	Description
10519	UVE™; Photochemical Reactor
1153-1098	PINNACLE PCX; single pump, 1,0 mL Reactor