

## N-NITROSODIETHANOLAMINE (NDELA) IN COSMETICS



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

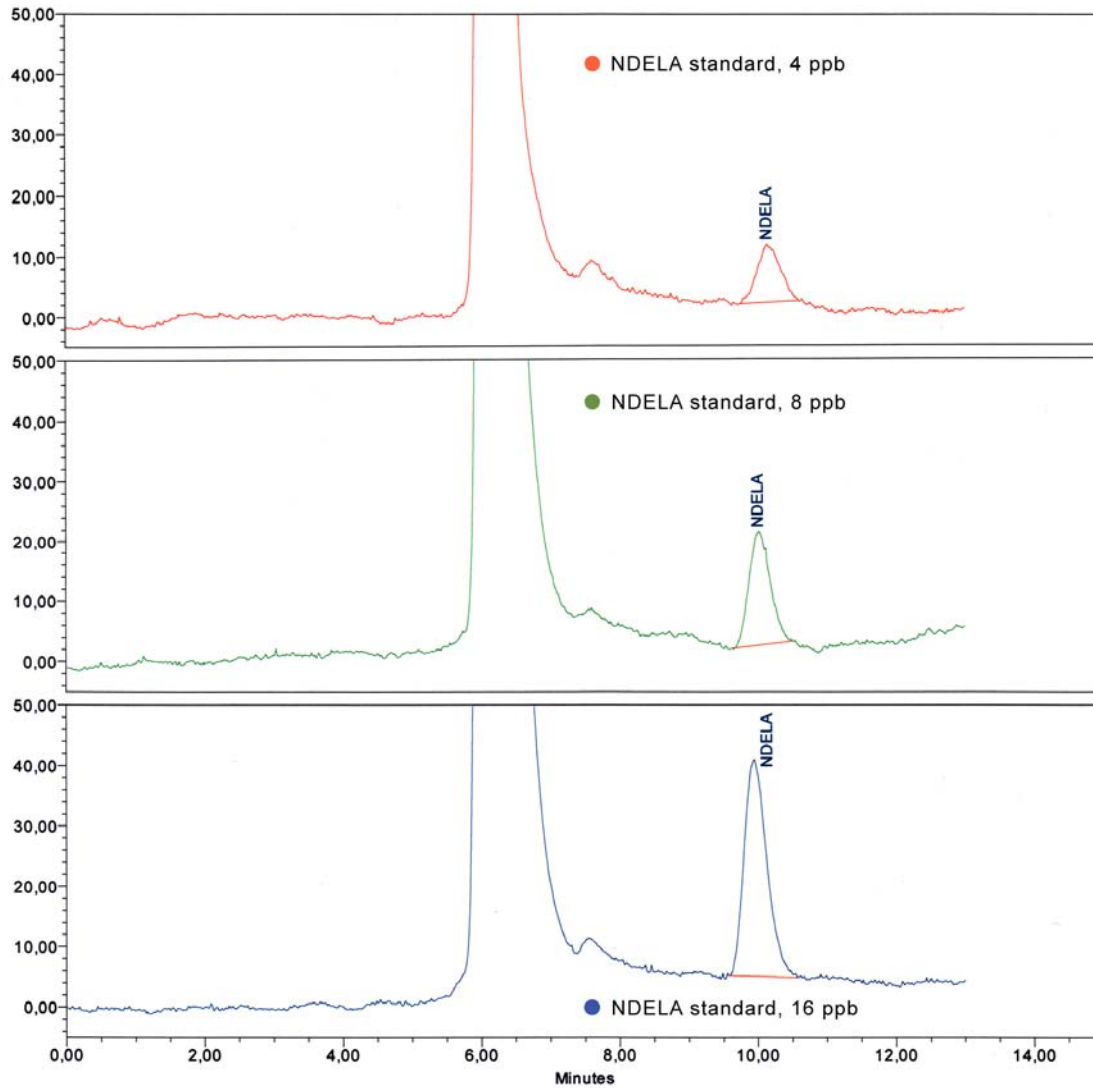
There are several analytical methods available, e.g. GC-TEA, HPLC-MS/MS or HPLC with photolysis and post-column derivatization. Mainly the last method is characterized by a high specificity, minimizes the risk of measuring artefacts and allows an accurate quantification.

Therefore this method is proposed by the Technical Committee for Cosmetics of the International Organization for Standardization. The procedure is described under DIN EN ISO 10130:2009.

### Description of Method

After a simple extraction and cleanup step using SPE or dichloromethane, respectively, NDELA is chromatographically separated from any remaining matrix compounds on an RP C18-HPLC column. Subsequently the N-nitroso-bond is photochemically cleaved to nitrite at 254 nm using the UVE™ photochemical derivatization module. In a second post-column step the formed nitrite reacts with the Griess-reagent in a single-pump post-column derivatization system PINNACLE PCX to yield a strongly colored azo dye which is detected at 540 nm.

## Chromatograms

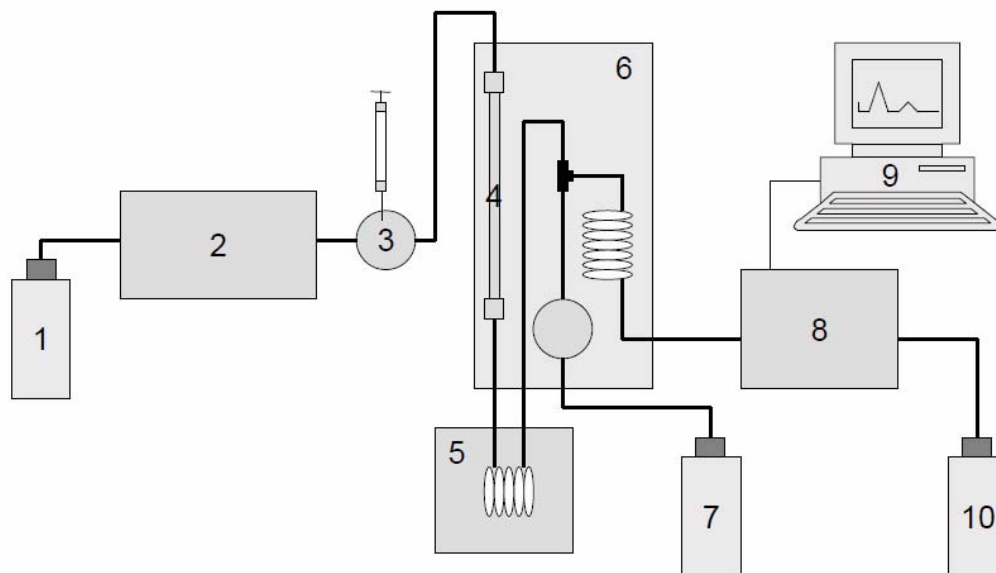


Chromatograms of NDELA standard: 4, 8 and 16 ppb  
Determination of N-nitrosodiethanolamine (NDELA) according to DIN EN ISO 10130:2009

## HPLC Conditions and Derivatization Parameters

HPLC Conditions	
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 pre-column 4.6 x 20 mm; C18, 5 µm
Column Oven	30 °C
Flow Rate	0.5 mL/min
Injection Volume	50 µL – 100 µL
Post-Column Derivatization	
UVE™	Photochemical Reactor
Pinnacle PCX	Single Pump
Reactor Volume	1.0 mL
Reactor Temperature	50 °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 light.
Reagent Flow	0.5 mL/min
Detection	
Detection Type	UV/VIS detection
UV/VIS	540 nm
Flow cell	Analytic; pressure stable up to 7 bar

## Flow Diagram



This flow scheme shows the typical configuration of an HPLC-instrument together with Pinnacle PCX and UVE™ for NDELA analysis.

- 1) HPLC eluant
- 2) HPLC pump
- 3) Sampling valve
- 4) HPLC column
- 5) UVE™ (LCTech GmbH, Germany) – photolysis unit: 254 nm
- 6) Pinnacle PCX post-column derivatization module (Pickering Laboratories, USA) including low dead-volume mixing tee, Griess reagent delivery pump, column oven, heated reactor coil (1mL; 50 °C)
- 7) Griess reagent
- 8) Detector (tungsten lamp) 540 nm
- 9) Computer
- 10) Waste

## Literature

1. DIN EN ISO 10130:2009  
Cosmetics - Analytical methods - Nitrosamines: Detection and determination of N-nitrosodiethanolamine (NDELA) in cosmetics by HPLC, post-column photolysis and derivatization.
2. A method for the determination of N-Nitrosodiethanolamine (NDELA) in Personal Care Products – Collaboratively evaluated by the CTPA Nitrosamines Working Group:  
Chris Flower, Stephen Carter, Andy Earls, Richard Fowler, Stewart Hewlins, Sam Lalljie, Mark Lefebvre, Jacqueline Mavro, David Small, and Nathalie Volpe

## Order Information

Order Number	Description
10519	UVE™; Photochemical Reactor
1153-1098	PINNACLE PCX; single pump, 1.0 mL Reactor