



VNIVERSITAT  
D VALÈNCIA

# DEVELOPMENT OF AN ANALYTICAL METHOD FOR THE DETERMINATION OF ACRYLAMIDE IN COSMETIC PRODUCTS BASED ON DISPERSIVE LIQUID-LIQUID MICROEXTRACTION

Lorenza Schettino\*, Juan L. Benedé, Alberto Chisvert\*, Amparo Salvador

Department of Analytical Chemistry, Faculty of Chemistry, University of Valencia (Spain)

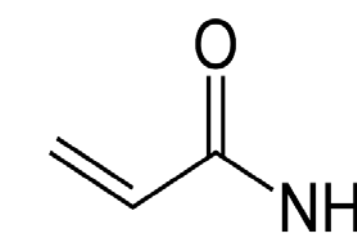
\*e-mail: los2@alumni.uv.es, alberto.chisvert@uv.es

gicapc  
grupo de investigación

## INTRODUCTION

The aim of this work is to develop and validate a new analytical strategy that allows to determine the presence of **acrylamide** (a mutagenic and potentially carcinogenic compound [1]) in cosmetic products, in order to establish if a cosmetic product respects the limit values as declared by the European Regulation

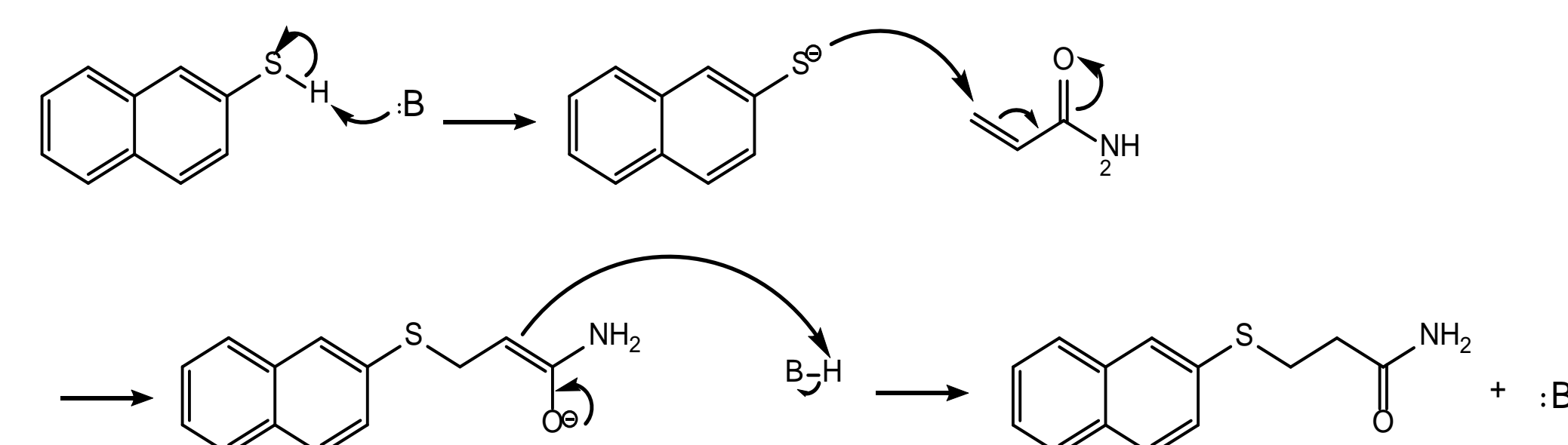
Acrylamide



The method is based on **dispersive liquid-liquid microextraction (DLLME)**, followed by liquid chromatography-ultraviolet (LC-UV) determination. In order to improve the selectivity and sensitivity of the analysis, a previous derivatization step was developed and optimized

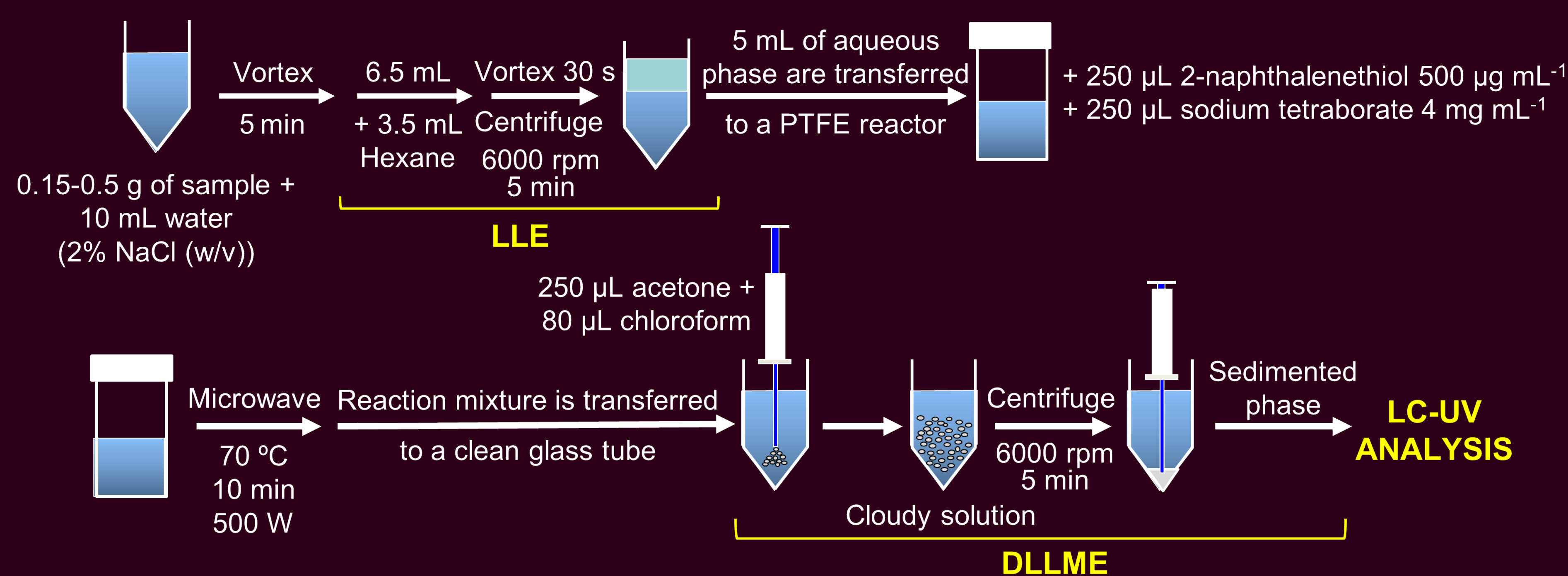
A **base-catalyzed Thiol-Michael Addition reaction** between acrylamide and 2-naphthalenethiol converts acrylamide into a less polar compound to be extracted by DLLME, and introduces a chromophore moiety to be analyzed by UV spectrometry detection

The **DLLME** is proposed to separate the analyte from possible interfering substances and to pre-concentrate it, in order to improve the limits of detection and quantification



The Base-Catalyzed Thiol-Michael Addition Reaction between acrylamide and 2-naphthalenethiol

## EXPERIMENTAL



Column	Purospher® RP-18 (125 x 4 mm, 5 µm)		
Inject volume	20 µL		
Temperature	Room temperature		
Flow rate	1 mL min <sup>-1</sup>		
Mobile phase	EtOH : H <sub>2</sub> O		
Gradient program	t (min)	EtOH (%)	H <sub>2</sub> O (%)
	0	50	50
	3	50	50
	4	80	20
	5	80	20
	5.5	50	50
7	50	50	
Wavelength	254 nm		

## RESULTS AND DISCUSSION

### Figures of merit of the proposed method

High level of linearity, that reached at least **100 ng mL<sup>-1</sup>**, was obtained

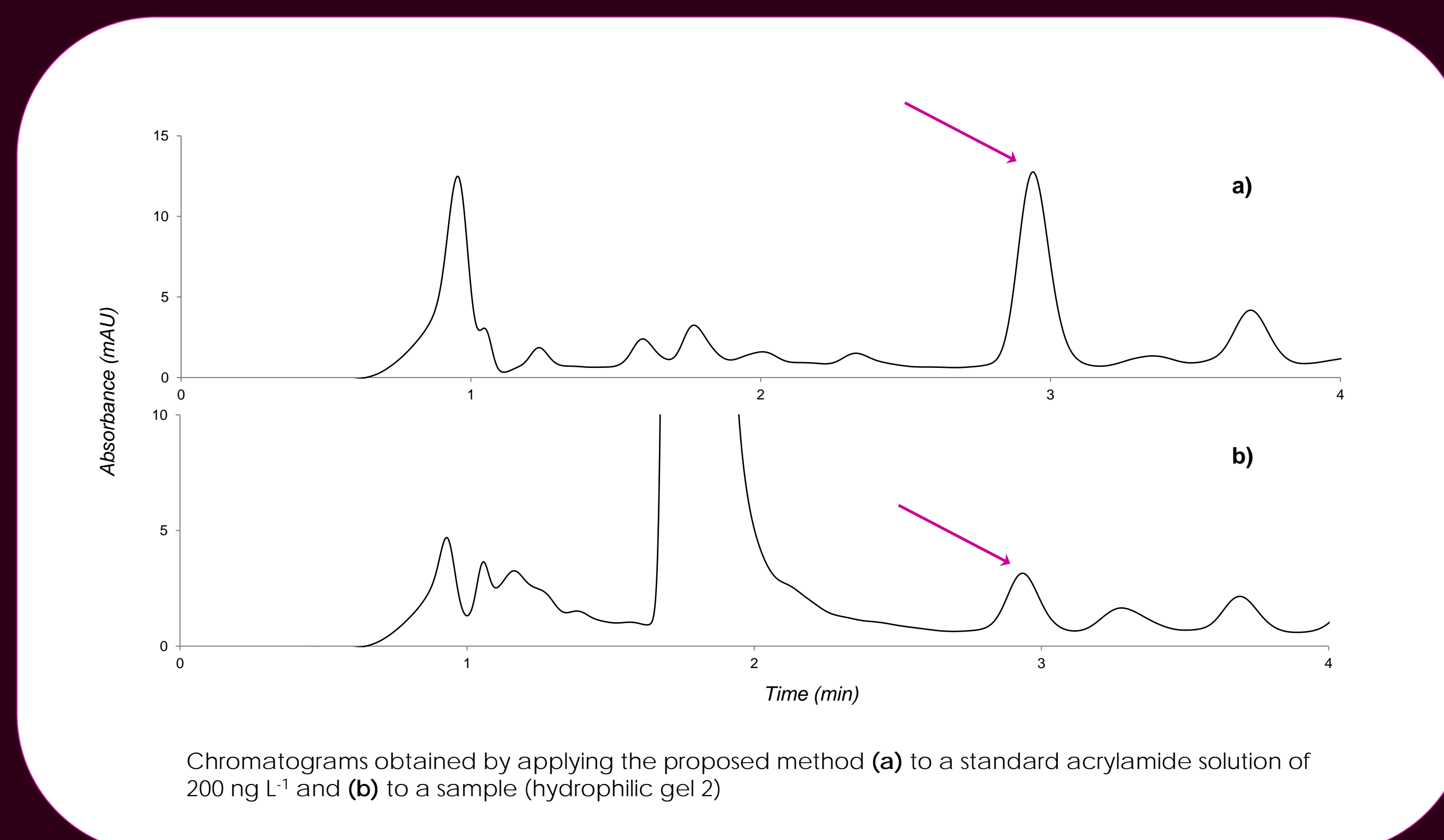
EF ± s	LOD <sup>a</sup> (ng mL <sup>-1</sup> )	LOQ <sup>b</sup> (ng mL <sup>-1</sup> )	MLOD (µg g <sup>-1</sup> )	MLOQ (µg g <sup>-1</sup> )	Repeatability (%RSD)			
					intra-day		inter-day	
					40 ng mL <sup>-1</sup>	80 ng mL <sup>-1</sup>	40 ng mL <sup>-1</sup>	80 ng mL <sup>-1</sup>
103 ± 2	3.0	9.8	0.03	0.10	8.9	3.7	13.5	8.1

<sup>a</sup>MLOD: Limit of detection of the method  
<sup>b</sup>MLOQ: Limit of quantification of the method

### Analysis of real samples (cosmetic products without rinsing)

Sample	Found amount (µg g <sup>-1</sup> )	Relative recovery (%)	
		0.4 ng g <sup>-1</sup>	0.8 ng g <sup>-1</sup>
Hydrophilic gel 1	2.3 ± 0.2	85 ± 11	100 ± 9
Hydrophilic gel 2	4.7 ± 1.1	80 ± 11	92 ± 3
Lipophilic cream	1.2 ± 0.1	80 ± 1	83 ± 3

The results of the recovery studies show that the matrices under consideration do not significantly affect the DLLME approach



Chromatograms obtained by applying the proposed method (a) to a standard acrylamide solution of 200 ng L<sup>-1</sup> and (b) to a sample (hydrophilic gel 2)

## CONCLUSIONS

The method allows to detect and quantify the concentration of acrylamide in cosmetic samples, of hydrophilic and lipophilic nature, with good analytical characteristics, such as accuracy, precision and sensitivity

It should be noted that the concentration of acrylamide detected in the samples exceeds the maximum limit values set in the European Regulation (limits established: <0.1 mg/kg for cosmetic products without rinsing and <0.5 mg/kg in other cosmetic products)



Consulta otras comunicaciones presentadas por el GICAPC en la **XXII Reunión de la Sociedad Española de Química Analítica:**

*Determination of nitro musks in environmental waters by stir bar sorptive dispersive microextraction followed by thermal desorption-gas chromatography-mass spectrometry.* J.L. Benedé, A. Chisvert, A. Salvador. **Flash Communication.** [Ver comunicación.](#)

*Stir bar sorptive-dispersive microextraction mediated by a magnetic nanoparticles-metal organic framework composite for the determination of n-nitrosamines in cosmetic products.* P. Miralles, I. Van Gemert, A. Chisvert, A. Salvador. **Flash Communication.** [Ver comunicación.](#)

*Development of an analytical method for the determination of acrylamide in cosmetic products based on dispersive liquid-liquid microextraction.* L. Schettino, J.L. Benedé, A. Chisvert, A. Salvador. **Flash Communication.** [Ver comunicación.](#)

*Determination of hydroxylated ingredients with preservative activity in cosmetic products by gas chromatography-mass spectrometry.* C. Azorín, J.L. Benedé, A. Chisvert, A. Salvador. [Ver comunicación.](#)

*A green analytical method for the determination of hydroxyethoxyphenyl butanone in cosmetic products.* P. Miralles, J.L. Benedé, A. Mata-Martín, A. Chisvert, A. Salvador. [Ver comunicación.](#)

*Determination of polycyclic aromatic hydrocarbons in cosmetics by stir bar sorptive dispersive microextraction and gas chromatography-mass spectrometry.* Váñez-Gomis, J. Grau, J.L. Benedé, A. Chisvert, A. Salvador. [Ver comunicación.](#)

*Reversed-phase dispersive liquid-liquid microextraction prior to liquid chromatography-tandem mass spectrometry for the determination of acrylamide in cosmetic products.* L. Fernández, J.L. Benedé, A. Chisvert, A. Salvador. [Ver comunicación.](#)

*Development of dispersive liquid-solid microextraction: application to the determination of cortisone and cortisol in human saliva.* J. Grau, J.L. Benedé, A. Chisvert, A. Salvador. [Ver comunicación.](#)