

REVERSED-PHASE DISPERSIVE LIQUID-LIQUID MICROEXTRACTION PRIOR TO LIQUID CHROMATOGRAPHY-TANDEM MASS SPECTROMETRY FOR THE DETERMINATION OF ACRYLAMIDE IN COSMETIC PRODUCTS

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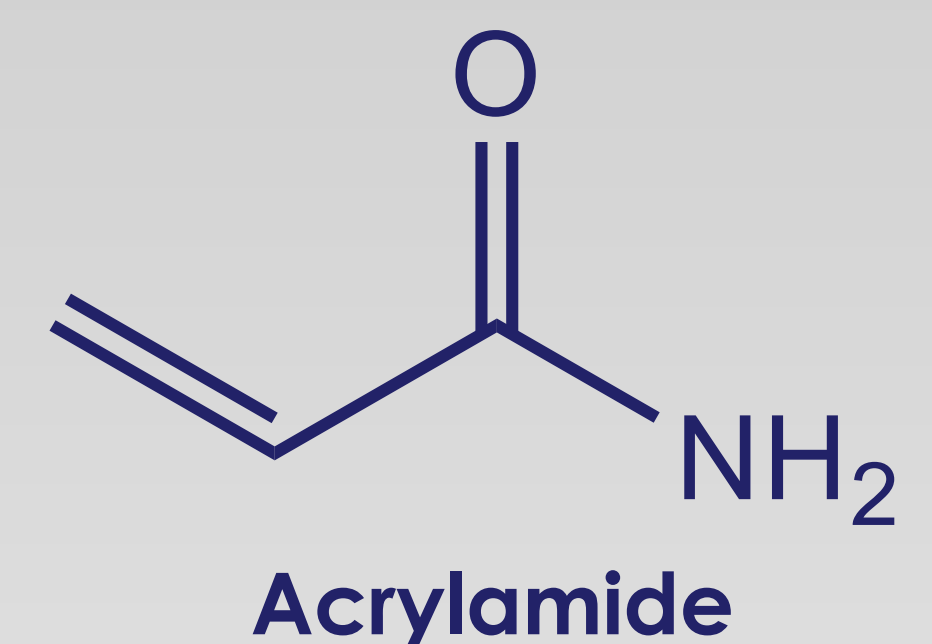
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INTRODUCTION

AIM

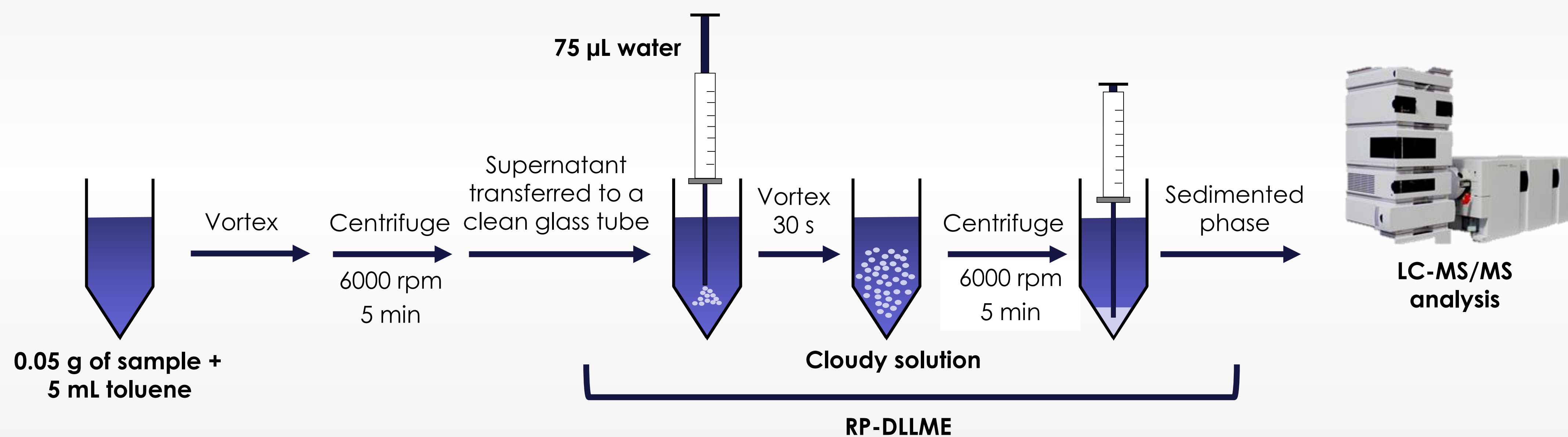
In this work, a fully optimized method for the determination of acrylamide in cosmetics products is presented. It is based on the high potential of the reversed-phase dispersive liquid-liquid microextraction (RP-DLLME) as clean-up and enrichment technique for highly polar and water-soluble analytes



Acrylamide-based polymers are cosmetics ingredients used as a stabilizing agents, antistatic agents, binders, film-formers, fixatives, conditioners, thickeners or rheological modifiers. Cosmetics products containing polyacrylamide might contain small amount of toxic **acrylamide** monomers, which have a high risk of causing cancer

The European Regulation prohibits the use of acrylamide as an ingredient in cosmetic products, and the use of polyacrylamide has been restricted, so that the maximum residual acrylamide content is **< 0.1 mg kg⁻¹** in cosmetic products without rinsing, and **< 0.5 mg kg⁻¹** in the rest of cosmetic products

EXPERIMENTAL



LC-MS/MS conditions

- **Injection Volume:** 5 µL
- **Column Temperature:** 30 °C
- **Mobile phase:** MeOH:H₂O(0.01% formic acid) (40:60%)
- **Flow:** 0.2 mL min⁻¹
- **Column:** Zorbax SB-C18
- **Gas Temperature:** 300 °C
- **Gas Flow:** 10 L min⁻¹
- **Acquisition mode:** ESI⁺ (MRM)
- **Capillary voltage:** +3500 V

RESULTS AND DISCUSSION

Figures of merit of the proposed method

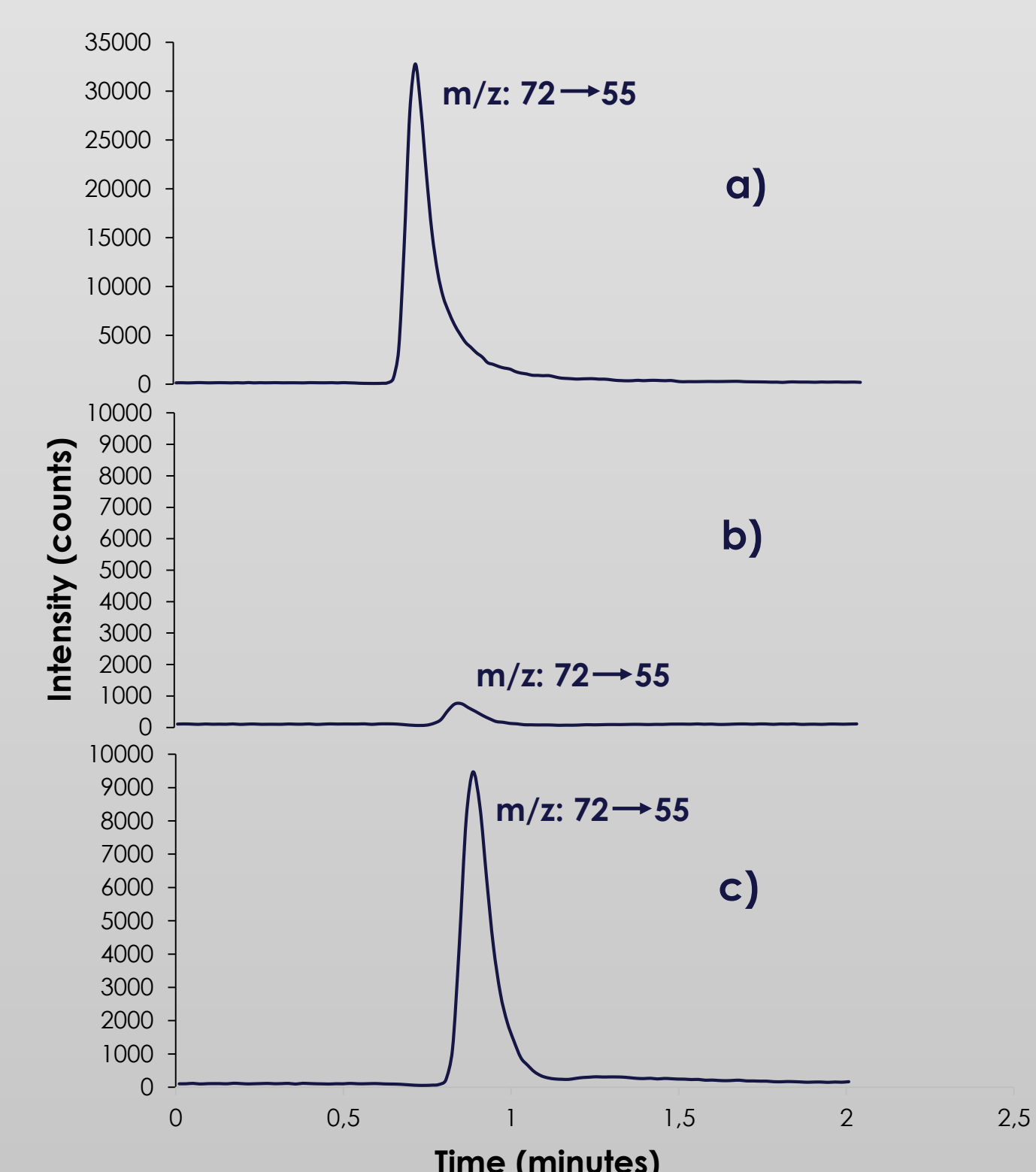
- High level of linearity, that reached at least **50 µg L⁻¹** was obtained
- Low **limits of detection** (0,13 µg g⁻¹) and good values of **precision** (< 10 %) and **enrichment factor** (30) were achieved

Analysis of real samples

	Refreshing gel		Hand cream		Hand soap	
	Found amount (µg g ⁻¹)	Relative recovery (%)	Found amount (µg g ⁻¹)	Relative recovery (%)	Found amount (µg g ⁻¹)	Relative recovery (%)
Acrylamide	< LOD	94 ± 3	0.048 ± 0.009	97.2 ± 0.4	0.384 ± 0.017	93.7 ± 0.8

- The results of the **recovery studies** (94 – 97 %) show that the matrices under consideration do not significantly affect the RP-DLLME approach

	LOD (µg g ⁻¹)	LOQ (µg g ⁻¹)	Enrichment factor	Repeatability (% RSD)			
				Intra-day		Inter-day	
				0.5 µg L ⁻¹	1 µg L ⁻¹	0.5 µg L ⁻¹	1 µg L ⁻¹
Acrylamide	0.13	0.45	30 ± 3	6.0	6.8	9.3	8.0



a) Chromatogram of a standard solution containing the analyte at 25 µg L⁻¹; b) Chromatogram of a real sample; c) Chromatogram of a fortified real sample at 25 µg L⁻¹

CONCLUSIONS

- Reversed-phase liquid-liquid microextraction is proposed for the first time to determine acrylamide in cosmetic products
- The method has been validated by obtaining quality analytical parameters, such as linearity, sensitivity, limits of detection and quantification, enrichment factor and relative recovery



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.](#)