DETERMINATION OF HYDROXYLATED INGREDIENTS WITH PRESERVATIVE ACTIVITY IN COSMETIC PRODUCTS BY GAS CHROMATOGRAPHY-MASS SPECTROMETRY

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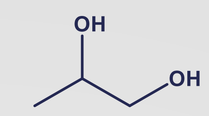
INTRODUCTION

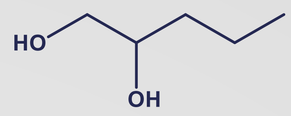
Some **hydroxylated compounds** commonly used in cosmetic formulations show antimicrobial activity, although they are not considered as preservatives according to the current European Regulation on Cosmetic Products [1].
The biologic activity of these **'alternative preservatives'** makes necessary their control to ensure quality and safety of the final product

There are no official methods to determine these compounds in cosmetic samples, and no procedures for the simultaneous determination of them have been described either

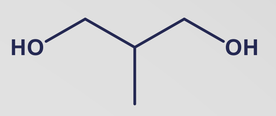
The **aim** of this work is to develop and validate an analytical method for the simultaneous determination of seven hydroxylated ingredients with preservative activity in cosmetic samples



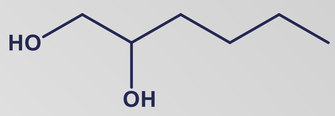
Propylene Glycol (PrG)



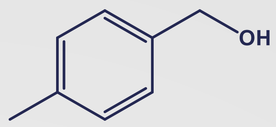
Pentylene Glycol (PtG)



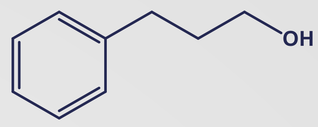
Methylpropanediol (MP)



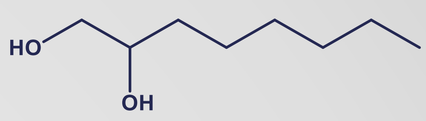
Hexylene Glycol (HG)



Methylbenzyl alcohol (MBA)



Phenylpropanol (PP)



Caprylyl Glycol (CG)

EXPERIMENTAL

0.01 g sample
+ Internal standard
(1-octanol)
EtOH up to 10 mL

Ultrasound assisted lixiviation

Filtration

GC-MS

GC-MS conditions

Inlet temperature:	230 °C
Transfer line temperature:	230 °C
Ion source temperature:	230 °C
Helium flow rate:	1 mL min^{-1}
Injection volumen:	2 µL
Column:	VF-WAXms

(polyethylene glycol, 30 m, 0.25 mm i. d., 0.25 µm)

RESULTS AND DISCUSSION

Figures of merit of the proposed method

- High level of **linearity**, that reached at least 10 µg mL^{-1}, was obtained for all compounds
- Low **limits of detection** and good values of **precision** were achieved

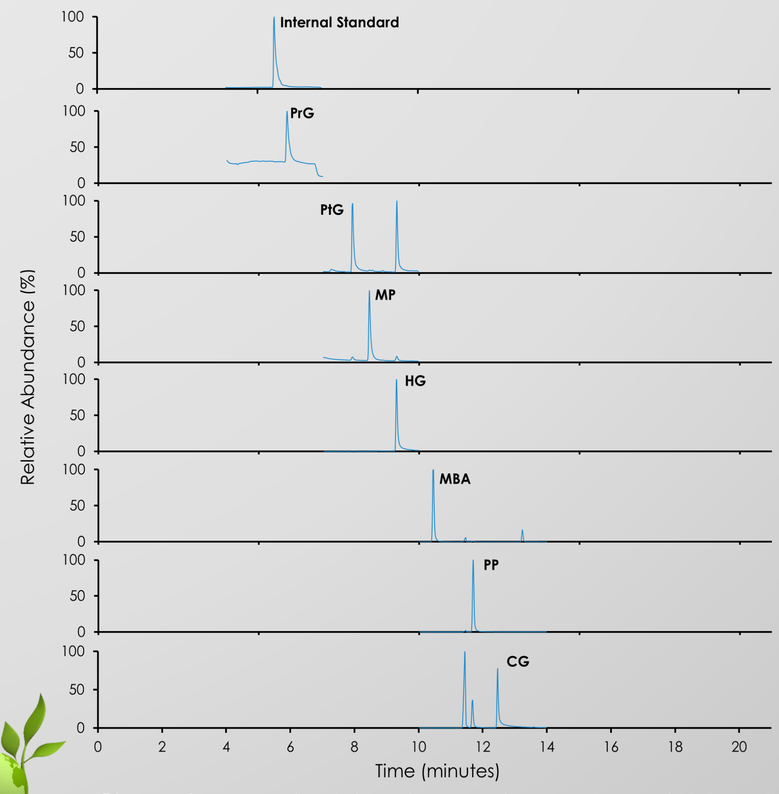
Analysis of real samples

The method was successfully applied to the analysis of eight commercial cosmetic samples and two laboratory-made samples, used to evaluate the accuracy of the method (relative errors where between 0.4 and 15.7 %)

Analyte	LOD (µg mL^{-1})	LOQ (µg mL^{-1})	MLOD (%, w/w)	MLOQ (%, w/w)	Repeatability (% RSD)			
					Intra-day		Inter-day	
					5 µg mL^{-1}	10 µg mL^{-1}	5 µg mL^{-1}	10 µg mL^{-1}
PrG	0.10	0.33	0.010	0.033	6	5	6	12
PtG	0.05	0.15	0.005	0.015	2	5	2	6
MP	0.05	0.15	0.005	0.015	6	5	13	11
HG	0.01	0.04	0.001	0.004	2	5	5	5
MBA	0.01	0.04	0.001	0.004	1	8	6	11
PP	0.02	0.06	0.002	0.006	6	7	12	10
CG	0.13	0.41	0.013	0.041	9	10	4	15

Sample	Found amount (Expected) (%)						
	PrG	PtG	MP	HG	MBA	PP	CG
Lab-made Cream	0.58 (0.54)	0.57 (0.54)	0.58 (0.50)	0.56 (0.54)	0.54 (0.50)	0.54 (0.50)	0.61 (0.54)
Lab-made Gel	1.76 (1.77)	0.50 (0.46)	0.53 (0.58)	0.43 (0.50)	0.43 (0.46)	0.48 (0.46)	0.46 (0.46)

Analyte	Relative recovery (%)							
	Refreshing Gel		Depigmenting Cream		Fluid Makeup		Liquid Soap	
	5 µg mL^{-1}	10 µg mL^{-1}	5 µg mL^{-1}	10 µg mL^{-1}	5 µg mL^{-1}	10 µg mL^{-1}	5 µg mL^{-1}	10 µg mL^{-1}
PrG	108 ± 10	108 ± 4	98 ± 5	97.5 ± 1.6	110 ± 5	98 ± 5	86 ± 6	82 ± 5
PtG	108 ± 4	114 ± 2	103 ± 8	96 ± 5	112.9 ± 1.7	119 ± 2	120 ± 8	112 ± 9
MP	98 ± 17	79 ± 14	87.2 ± 1.9	92 ± 3	130 ± 2	124 ± 6	124 ± 8	106 ± 9
HG	113 ± 7	112 ± 3	100 ± 4	96 ± 6	107 ± 8	100 ± 5	103 ± 5	102 ± 4
MBA	107 ± 7	102 ± 6	93 ± 8	84.3 ± 1.9	114 ± 6	106 ± 6	119 ± 7	107 ± 8
PP	99 ± 8	94 ± 2	77 ± 9	68 ± 3	97 ± 20	93 ± 10	125 ± 7	117 ± 9
CG	105 ± 15	113 ± 9	110 ± 30	92 ± 12	104 ± 20	116 ± 6	115 ± 7	119 ± 2



Chromatogram of a laboratory-made sample containing all the analytes of interest

CONCLUSIONS

- The developed method constitute an **efficient** and **reliable** procedure with good **analytical features** that could be easily applied to the quality control of cosmetic products
- In addition, the method is in accordance with the principles of the **'Green Analytical Chemistry'**, as it is harmless to the operator and the environment

[1] Regulation (EC) No. 1223/2009 of the European Parliament and of the Council of 30 November 2009 on cosmetic products.

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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. Vállez-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.