

Quantative analysis of allergens in cosmetics with GC/MS

Key words:

OPTIC
Allergens
Cosmetics
Quantative

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Introduction

In 2003, regulation (EU) was introduced to regulate the usage of fragrances which are considered allergenic. 1-2% of the population seems to be allergic. To minimize contact allergic reaction the threshold where set to 0.01% for a rinse-off product (shampoo) and 0.001% for leave-on products (hand cream).

Because of this regulation, manufacturers require dedicated instruments for quantification of these allergens.

Sample materials

3 standard of allergens, concentrations, 10, 40, 100 ng/μl. (injection volume = 1 μl)

8 raw samples, diluted with methanol 10x.

Cosmetic cream, body oil, cleaning product and shower gel.

Equipment

PTV Injector: OPTIC 3

Liner: L100011, DMI liner with taper

Auto sampler: CombiPAL

GC-MS: Shimadzu QP2010

GC column: : InertCap 5 MS/Sil, GL Sciences, 0.25 m x 30 m, film 0.5 μm

Methods

OPTIC method:

Method Type	Split
Method Name	Allergenes
Liner	L100011, DMI liner
Equilibration Time	00:05 mm:ss
End Time	29:30 mm:ss
Initial Temperature	35 °C
Ramp Rate	5.0 °C/s
Final Temperature	250 °C
Hold Time	05:00 mm:ss
Temperature Control	Floating Temperature
Carrier Control Mode	Flow Control
Transfer Time	03:00 mm:ss
Transfer Column Flow	1.0 ml/min
Start Column Flow	1.0 ml/min
End Column Flow	1.0 ml/min
Split Flow	100 ml/min

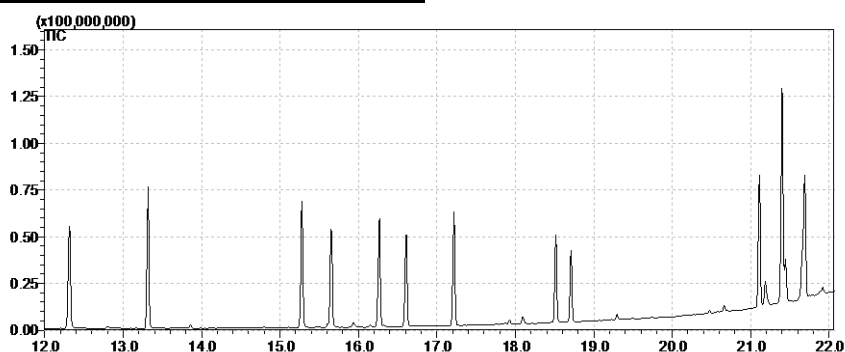
GC method

Ramp rate (°C/min)	Set Temperature (°C)	Hold time (min)
-	35.0	3.00
10.00	250.0	5.00

MS method

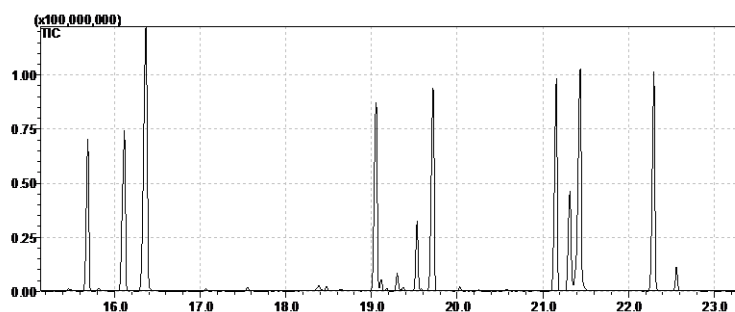
Begin time	End time	Mode	Scan length (sec)	Scan speed	Begin m/z	End m/z
3.00	29.50	Scan	0.50	666	35.00	350.00

Results Standard samples A, B, C

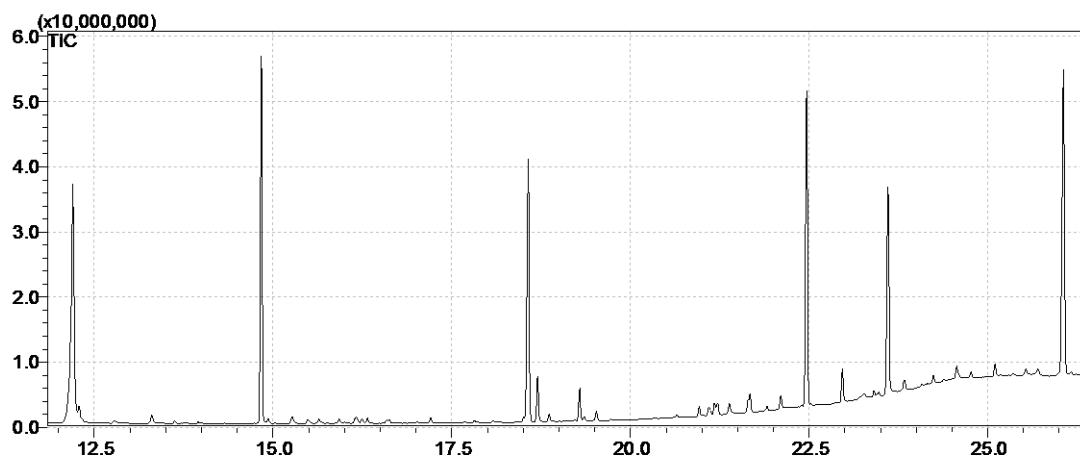


Sample A, Alcohols Solution consists out of

Name component	Ret Time (min)
Benzyl Alcohol	12.312
Linalool	13.317
Citronellol	15.279
Geraniol	15.654
Anisyl Alcohol	16.264
Cinnamyl alcohol	16.607
Eugenol	17.215
Isoeugenol	18.513
Amylcinnamyl alcohol	21.113
Farnesol	21.404
Farnesol	21.696
Cinnamaldehyde, .alpha.-hexyl-	22.113


Sample B, Aldehydes and Cetones Solution consists out of

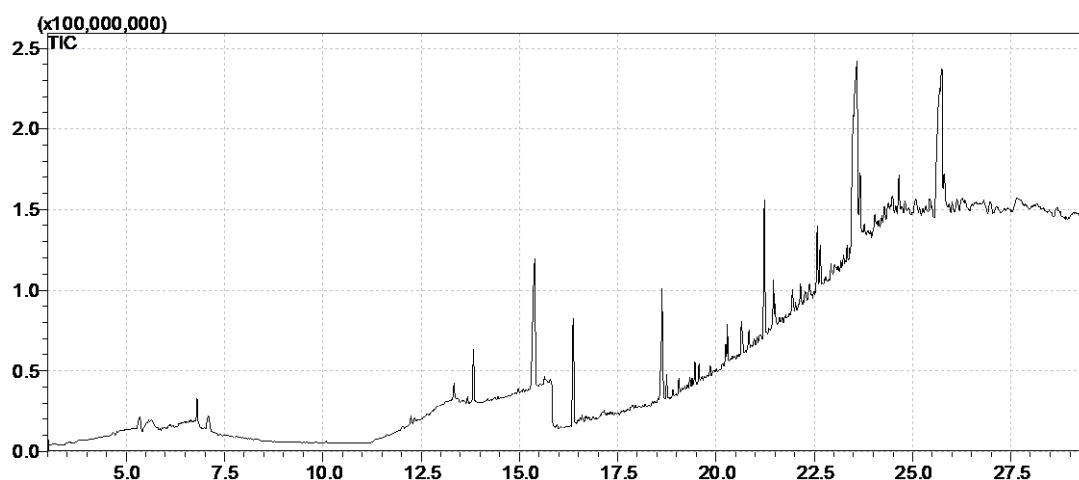
Name Compound	Ret Time (min)
Citral	15.511
Citral	15.926
Citronellal, hydroxy-	16.333
.alpha. Isomethyl ionone	18.871
Lilial	19.531
Cinnamaldehyde, .alpha.-pentyl-	20.973
Amylcinnamyl alcohol	21.110
Lylal	21.111
Lylal	21.238
Cinnamaldehyde, .alpha.-hexyl	22.399



Sample C, Esters Solution consists out of

Name Compound	Ret Time (min)
D-Limonene	12.198
Methyl 2-octynoate	14.832
Coumarin	18.562
Benzyl Benzoate	22.464
Benzyl salicylate	23.602
Benzyl cinnamate	26.051

Results Raw sample
Cream based product



Name Compound	Ret Time (min)	m/z	Area	RSD's	Amount (µg/ml)
D-Limonene	12.241	68.00	2525392	8%	30.80
Citral	15.639	41.00	725549	7%	12.92
Geraniol	15.683	69.00	696831	9%	7.02
Citral	15.954	69.00	282420	5%	6.13
Cinnamyl alcohol	16.634	92.00	51418	5%	1.07
Eugenol	17.246	164.00	101188.00	6%	1.60
.alpha. Isomethyl ionone	18.904	135.00	1563043	9%	13.05
Lilial	19.564	189.00	4009084	5%	40.10
Cinnamaldehyde, .alpha.-pentyl-	21.000	129.00	370996	6%	7.09
Cinnamaldehyde, .alpha.-hexyl-	22.145	129.00	3144191	1%	63.36
Benzyl salicylate	23.648	91.00	13512534	3%	67.06

Conclusion:

OPTIC is very suitable for the analysis of allergens with limited sample preparation. The high boiling compounds remain in the liner, after a limited transfer of the compounds of interest.

And of course, a minim of sample preparation. Only diluting the product with 10x methanol.

With RSD's lower than 10%, this analysis is very suitable to be used as a quantifying method.