

Application Data Sheet

No.37

GC-MS

Gas Chromatograph Mass Spectrometer

GC/MS Quantitative Analysis of Allergens in Cosmetics Using OPTIC-4

Because an estimated 1 to 2 percent of the population has allergies, in 2003 the EU began regulating the use of perfumes and flavour materials that can cause allergic reactions. These substances are restricted to 0.01 % for cosmetic products that are rinsed away (such as shampoo) and 0.001 % for products that are left applied (such as hand creams). Due to these regulations, manufacturers now must quantitatively analyze the concentration of such allergens.

Experiment

Sample

Standard samples of allergen mixtures and a cosmetic cream diluted in solution by ten times were analyzed.

Analysis Conditions

Sample

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Table 1 Analysis Conditions

Instruments

Inlet: OPTIC-4 (ATAS GL International BV)
 Liner: L100011, DMI liner with taper (ATAS GL International BV)
 GC-MS: GCMS-QP2010 Ultra (Shimadzu Co.)
 Autosampler: AOC-5000 Plus (Shimadzu Co.)
 LINEX (ATAS GL International BV)
 Column: InertCap 5 MS/Sil 30m x 0.25 mm I.D., df=0.5 µm (GL Science)

Inlet

Injection mode : Split
 Balancing time : 5sec
 Analysis time : 29.5min
 Vaporization chamber temperature
 : 35°C → (5°C/min) → 250°C(5min)
 Carrier gas : Helium
 Control mode : Flow control
 Transfer time : 3min
 Column flow rate: 1.0 ml/min
 Split flow : 100 ml/min

[MS]

Interface temperature: 250°C
 Ion source temperature: 200°C
 Solvent elution time: 3 min
 Data sampling time: 3 – 29.5 min
 Measurement mode: Scan
 Mass range: *m/z* 35-350
 Detector voltage: Relative value
 mode

[GC]

Column oven temperature:
 35°C(3min) → (10°C/min) → 250°C(5min)

Results and Discussion

Standard Sample Measurement

Figures 1, 2 and 3 show the results of measuring 3 standard samples.

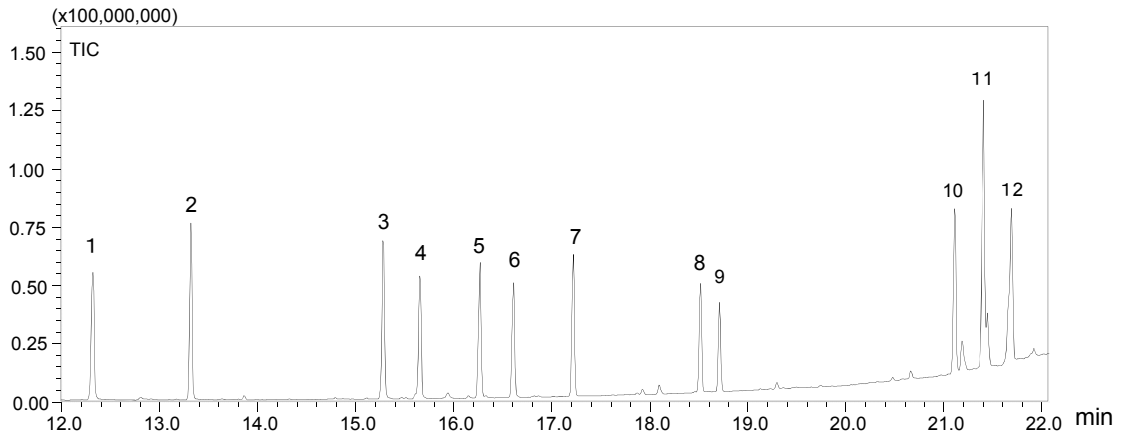


Fig. 1: Measurement Result of Allergen Standard Sample A

1.Benzyl Alcohol, 2.Linalool, 3.Citronellol, 4.Geraniol, 5.Anisyl Alcohol, 6.Cinnamyl alcohol, 7.Eugenol, 8.Isoeugenol, 9.Amylcinnamyl alcohol, 10.Farnesol, 11.Farnesol, 12.Cinnamaldehyde, .alpha. - hexyl -

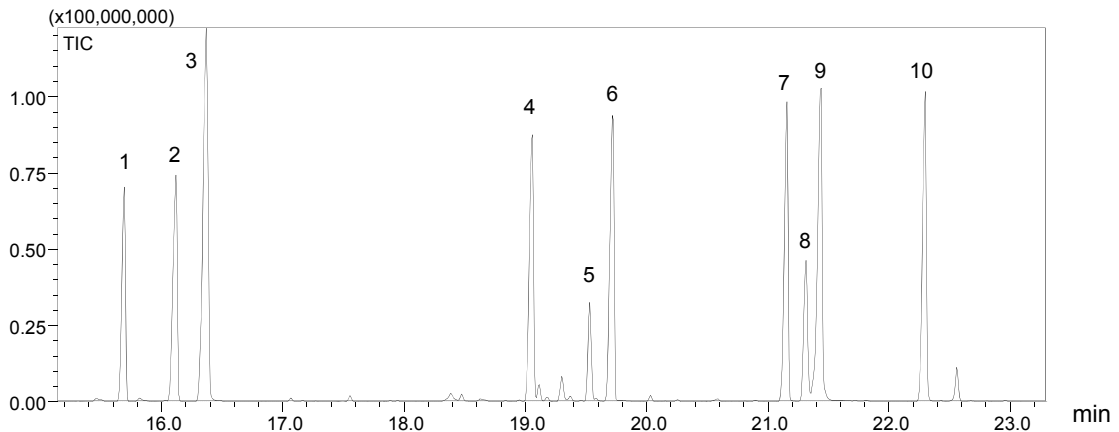


Fig. 2: Measurement Result of Allergen Standard Sample B

1.Citral; 2.Citral; 3.Citronellal, hydroxy - ; 4. alpha.Isomethyl ionone; 5.Lilial; 6. Cinnamaldehyde, .alpha. - pentyl - 7.Amylcinnamyl alcohol; 8.Lyral (21.111); 9.Lyral (21.238); 10.Cinnamaldehyde, .alpha. - hexyl

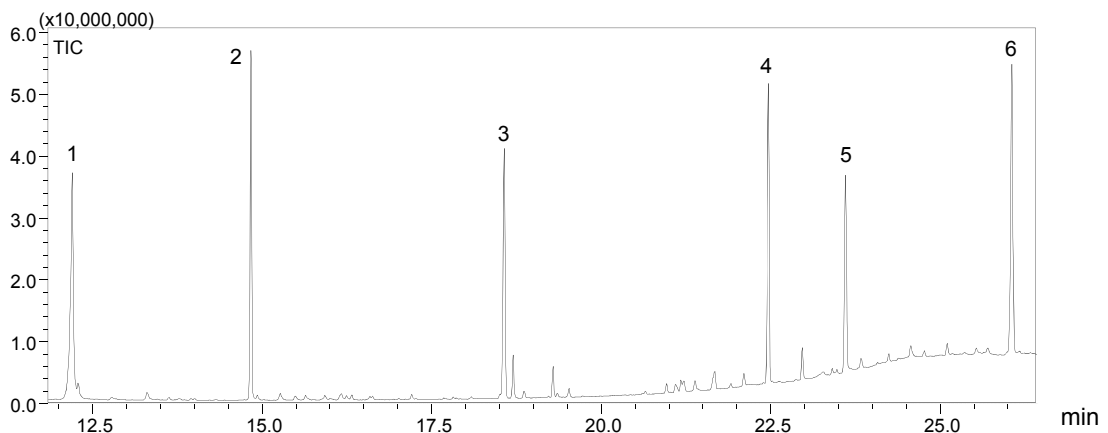
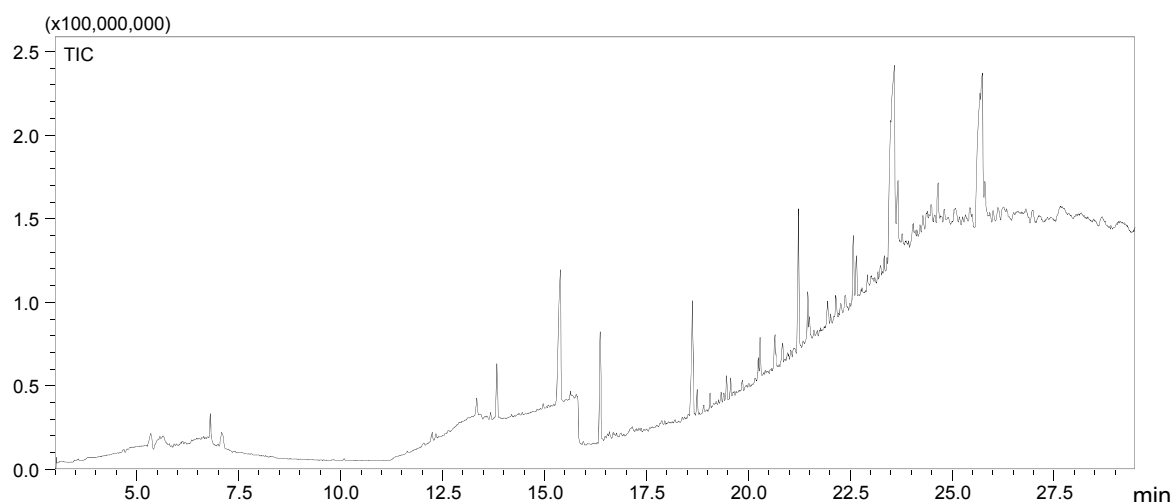


Fig. 3: Measurement Result of Allergen Standard Sample C

1.D - Limonene; 2.Methyl 2 - octynoate ; 3.Coumarin; 4.Benzyl Benzoate; 5.Benzyl salicylate; 6.Benzyl cinnamate

Actual Sample Measurement

Cosmetic cream measurement results are shown in Figure 4.



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

Fig. 4: Cosmetic Cream Measurement Results

The total ion chromatogram and area and repeatability accuracy of detected components are indicated above.

Summary

By using a DMI liner and OPTIC-4 inlet to adjust the injection port temperature, components targeted for measurement can be introduced to the column, while unwanted high boiling point substances are left remaining in the micro vial inside the DMI liner. Consequently, allergens can be analyzed by simply diluting measurement samples with methanol to 1/10 concentration. Measuring the allergens in cosmetic cream resulted in repeatability and %RSD values of 10 % or less, which indicates that the given method can be used to quantitatively analyze allergens.

This Application Datasheet was prepared in cooperation with Erwin Kaal and Iwan Horsting at ATAS GL International B.V.