Application Note No. 106

The Determination of Allergens in Cosmetics using GCXGC(qMS)

Key Words: DMI, Allergens, Cosmetics, GCxGC

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Introduction:

The quantitative determination of potential allergen compounds defined by the international fragrance association (IFRA) in cosmetics (perfumes, crèmes etc) is a complex analytical task due to possible interference with matrix peaks in the chromatogram which could lead in false positive or false negative identification. One approach to solve that problem is using comprehensive GCxGC(qMS) using rapid scanning GCMS equipment with low interscan delay. For products like crèmes, lipstick, shower gels or even for perfume control of fabric softeners the direct thermal desorption from the matrix was done by using the concept of difficult matrix introduction (DMI) with the OPTIC 3 injector in combination with comprehensive GCxGC(qMS) (GCMS-QP2010 Plus, Shimadzu) using a cryogenic modulator (ZOEX corporation, USA).

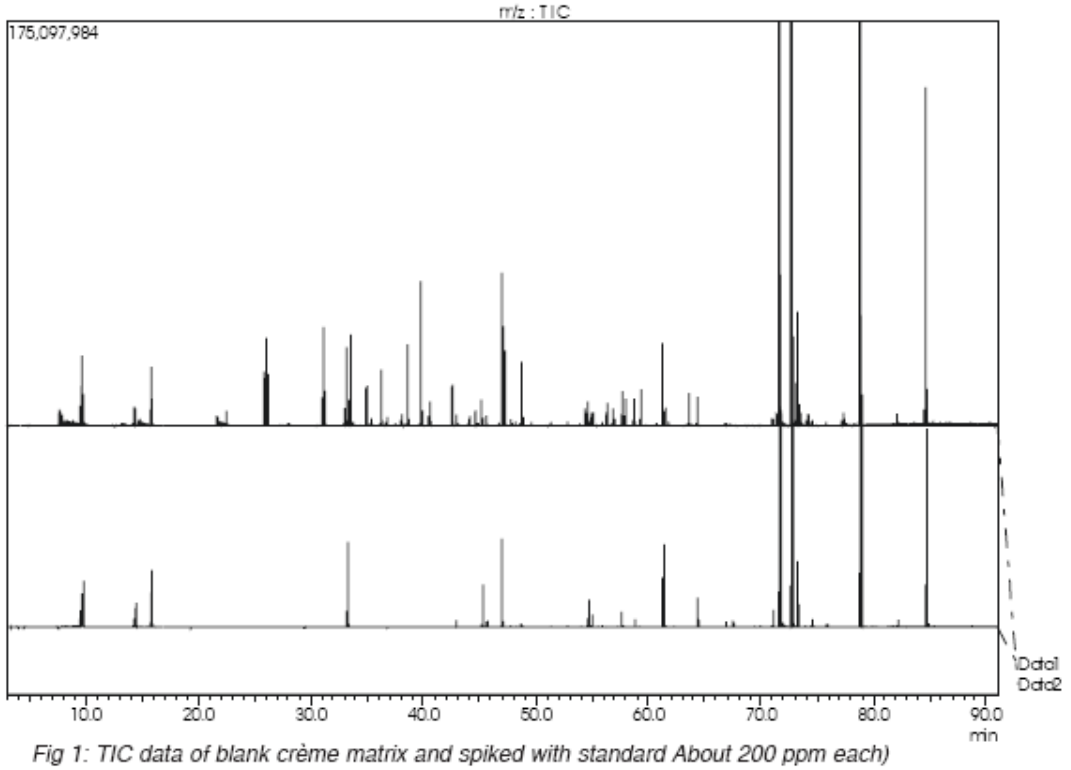


Fig 1: TIC data of blank crème matrix and spiked with standard About 200 ppm each)

Matrices like crèmes can be placed directly into a microvial and desorbed by an appropriate temperature program. Here a programme of 45 °C, 16 °C/sec to 280 °C was used. To have orthogonality a RTX-1, 30 m, 0.25 mm, 0.25 µm was used in the first dimension while a WAX column of 1 m, 0.1 mm, 0.1 µm was used in the second dimension. The loop consisted of 1.6 m (WAX). The quadrupol detector were operated in full scan mode with a mass range of 50- 200 amu and a sampling frequency of 50 Hz. The GC program selected was 50 °C, 1 min, 2.5 °C/ min to 250 °C. The split ratio was set 250:1. The modulation frequency was set to 8 s. The modulated peaks had a width of about 100 msec (FWHM). This is shown as an example for a standard sample containing 24 potential allergens (figure 1, table 1). The zoom (figure 2) shows methyl heptin carbonate and across such a peak using the above scan range 14 data points were acquired which is enough for quantitative work. The calibration for crème matrices was performed by using blank matrix which was spiked with the allergen standard. The regression coefficients were better than R = 0.99. Figure 3 shows the image created from a hand creme sample where 3 potential allergens were identified. The concentrations are listed in table 1.

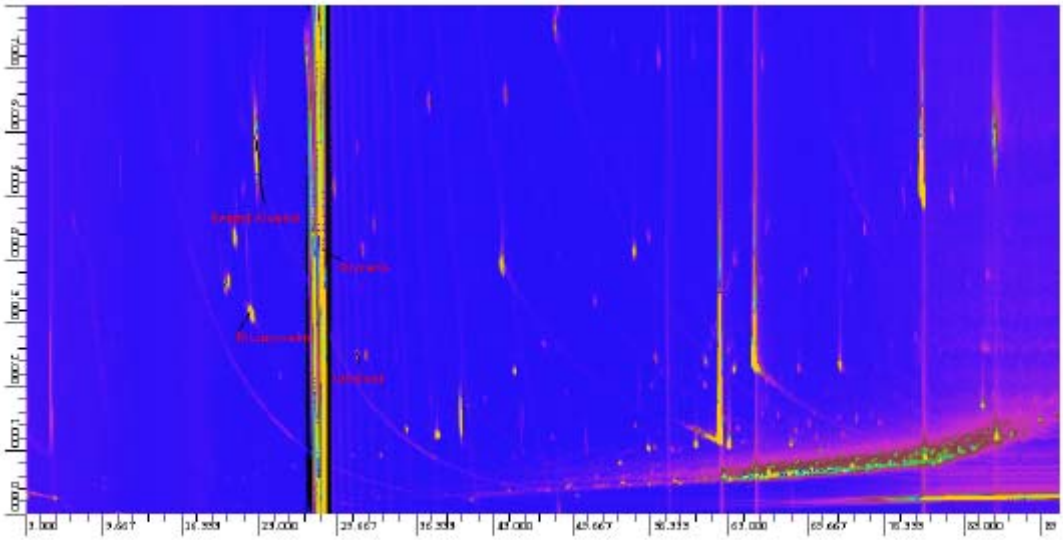
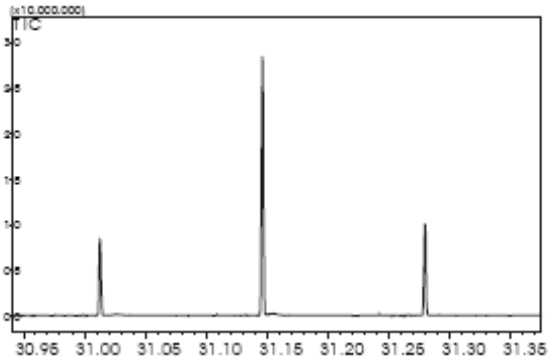


Fig.3: Image created from data recorded with a hand crème sample (1.4 mg)



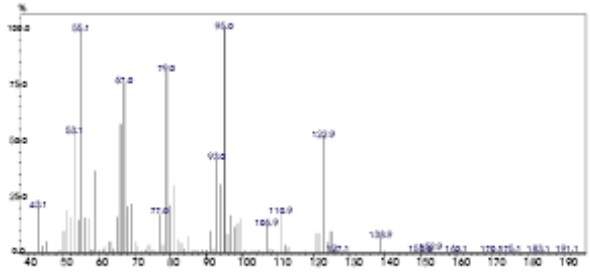


Fig 2: TIC and spectrum of methyl heptin carbonate

The spectra qualities over the narrow modulated peaks are very good proved by similarity indices SI larger than 95. For identification in addition the concept of linear retention index may be used also in comprehensive GCxGC(qMS). A special library for Flavour and fragrances is available (FFNSC 1.2, Shimadzu Europa GmbH)

Conclusion

DMI and comprehensive GCxGC(qMS) is an approach to overcome separation problems in complex samples.

Allergens	Hand crème (ppm)	Allergens
1. Benzyl Alcohol	108	13.Coumarin
2. Limonene	95	14.Iso-Eugenol
3. Linalol	77	15.Methyl Gamma Ionone
4. Methyl HeptinCarbonate		16.Lilial
5. Citronellol		17.Amyl Cinnamic Aldehyde
6. Citral		18.Lyral
7. Cinnamic Aldehyde		19.Amyl Cinnamic Alcohol
8. Geraniol		20.Farnesol
9. Anisic Alcohol		21. Hexyl Cinnamic Aldehyde
10. Hydroxy Citronellal		22. Benzyl Benzoate
11. Cinnamic Alcohol		23. Benzyl Salicylate
12. Eugenol		24. Benzyl Cinnamate

Table 1: List of Allergens

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