Quantitative Analysis of Allergens in Cosmetics Using Comprehensive Two-Dimensional GC and Time-of-Flight Mass Spectrometry (GC×GC–TOF-MS)

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Introduction

Complex fragrance mixtures are a part of everyday life. From cosmetics to household products, chemicals that produce scent are present at various levels, but little is known about their composition because fragrance formulas are considered trade secrets. The 7th Amendment of the European Cosmetics Directive requires the declaration of the listed 26 "fragrance allergens" (24 defined volatile substances and two botanicals) if they exceed specified levels.

This study addresses the detection of allergens in cosmetic products of the compounds targeted by the European Cosmetic Directive. A Pegasus 4D GC×GC–TOF-MS (LECO, Saint Joseph, MI, USA) system acquiring data at 200 spec/sec was used to identify and quantify the presence of the target compounds. Data processing was performed using ChromaTOF software (LECO).

Experimental

GC: Agilent 7890 with split/splitless injector and LECO quad-jet dual stage cryogenic modulator and secondary oven
Primary column: Rxi-5Sil MS, 30 m × 0.25 mm × 0.25 µm
Seconday column: Rtx-WAX, 1 m × 0.1 mm × 0.1 µm
Primary oven program: 90 °C (1 min) – 5 °C/min – 235 °C
Secondary oven program: 100 °C (1 min) – 5 °C/min – 245 °C
Modulation period: 10 seconds
Mass range: 35 – 350 m/z
Acquisition rate: 200 spectra/s

Figure 1: Calibration curve of hexylcinnamaldehyde.

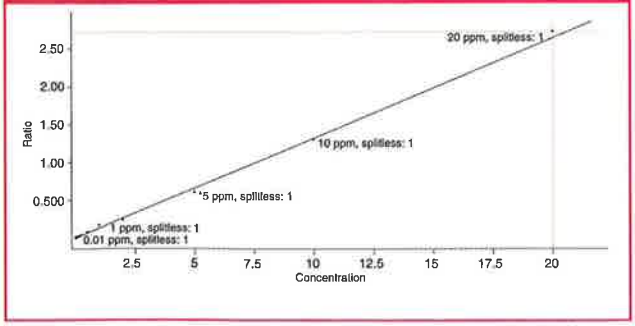
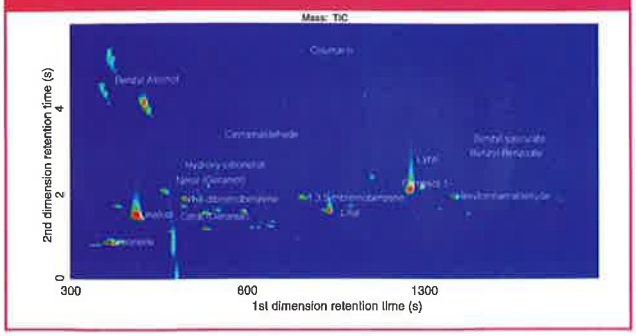


Figure 2: Contour plot of a typical shampoo.



Calibration

Standards at nine levels, 0.1 – 20 ppm in MTBE, were used to construct calibration curves. Correlation coefficients were 0.965–0.998. Two internal standard compounds (1,3-dibromobenzene, 1,3,5-tribromobenzene) were used at the level of 10 ppm. Figure 1 shows the calibration curve of Hexylcinnamaldehyde.

Quantification

The constructed calibration tables were used for automated quantitative screening of unknown samples. Figure 2 shows the contour plot of a typical shampoo 10 times diluted with MTBE prior to injection. The target compounds were automatically searched, identified and quantified. Without separation by GC×GC coelutions of target analytes with matrix compounds will occur, making data processing very difficult and time consuming. ChromaTOF's Automatic Peak Finding and Deconvolution algorithm accomplishes the difficult task, delivering pure and full range mass spectra for analyte identification.

Conclusions

This work demonstrates the use of GC×GC–TOF-MS for automated and fast target screening of allergenic compounds in cosmetics. This is an innovative approach demonstrating a number of advantages over other strategies of GC–MS analysis.

The power of Pegasus 4D GC×GC–TOFMS lies in its separation power combined with automatic data handling. Peak finding, deconvoluted spectrum determination and quantification are all automatically performed. This is all possible due to the high degree of spectral continuity as well as the large data density provided by the fast acquisition rate of LECO TOF-MS technology.

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