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Everything flows – crystalline and fluid structures in HPLC

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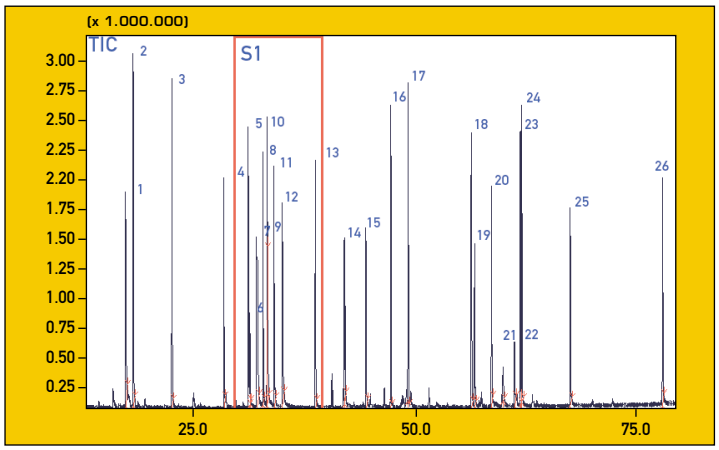

Figure 1: Total Ion Chromatogram (TIC) of an allergen standard (approx. 400 ppm, 26 compounds). Linear Velocity 34.4 cm/s (He), 50 °C 1 min, 2 °C/min up to 210 °C, 10 °C/min up to 280 °C. Split ratio 50 : 1

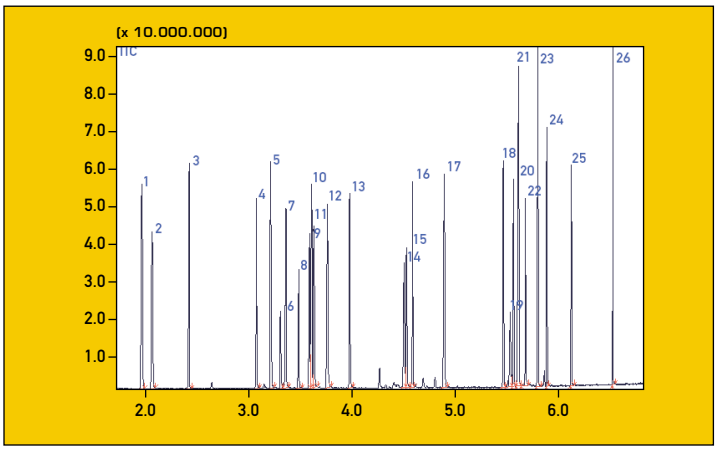

Figure 2: Allergen Mix measured with an SPB5 10 m, 0.1 mm ID, 0.1 µm Linear Velocity 40 cm/s (He), Split ratio 300 : 1, Temperature Ramp 70 °C 1 min, 25 °C/min up to 180 °C, 80 °C/min up to 280 °C 1 min. Mass range 30 - 350 amu 20 spectra/s (10.000 amu/s)

thing in the air

ermination of allergens in perfumes

The analysis of allergen compounds has become more and more important as allergenic-induced diseases in human beings has increased drastically in recent years.

In particular, allergic reactions to scent of perfumes is a topic of increasing research. The problem arises from the fact that the compounds of a specific perfume can induce an allergic reaction showing differing symptoms between individual human beings. However, the analysis is very complex and time consuming. Usually this is done by using quadrupole GCMS equipment. As an example, the result of an analysis recorded with an allergen standard is plotted in figure 1.

The concentration of each compound was approximately 400 ppm with 26 compounds in total, separated on a CP SIL 5 50 m column with 0.25 mm ID and 0.25 µm film thickness. Objective is to reduce the analysis time of 75 minutes in order to enhance the efficiency of the equipment. However, analysis time cannot be reduced by changing temperature and pressure parameters using a given column without affecting the resolution.

In order to find a way of reducing analysis and cycle time while maintaining resolution, narrow bore columns have become more and more useful in increasing efficiency of analysis in different fields [1-3].

The van Deemter curves of columns with reduced inner diameter result firstly in smaller HETPmin (Height equivalent of theoretical plates) values which approach the inner diameter of the columns, and secondly have smaller slopes above the HETP minimum so that the linear velocity of the carrier gas <u> can be raised above the optimum by a factor of 2 or 3 without significant loss of resolution. This results in a drastic increase of separation efficiency as it increases in inverse proportion to the HETP value [3]. When using these columns, the instrument hardware has to fulfil some minimum requirements. These are fast sample transfer to the column, high dynamic range of carrier gas pressure and high linear temperature ramps [4].

To run the columns at optimum separation efficiency for different temperatures, the GC component should also be able to maintain mean linear velocity of the D

Description figures:

1 Limonene
2 Benzyl Alcohol
3 Linalool
4 Methyl Heptin Carbonate
5 Citronellol
6 Neral
7 Geraniol
8 Citral
9 Cinnamic Aldehyde
10 Hydroxy Citronellal
11 Anisyl Alcohol
12 Cinnamyl Alcohol
13 Eugenol
14 Isoeugenol 1
15 Coumarin
16 6-Methyl-Gamma-Ionone
17 Lilial
18 Amyl Cinnamic Aldehyde
19 Farnesol 1
20 Lyral
21 Amyl Cinnamic Alcohol
22 Farnesol 2
23 Hexyl Cinnamic Aldehyde
24 Benzyl Benzoate
25 Benzyl Salycilate
26 Benzyl Cinnamate

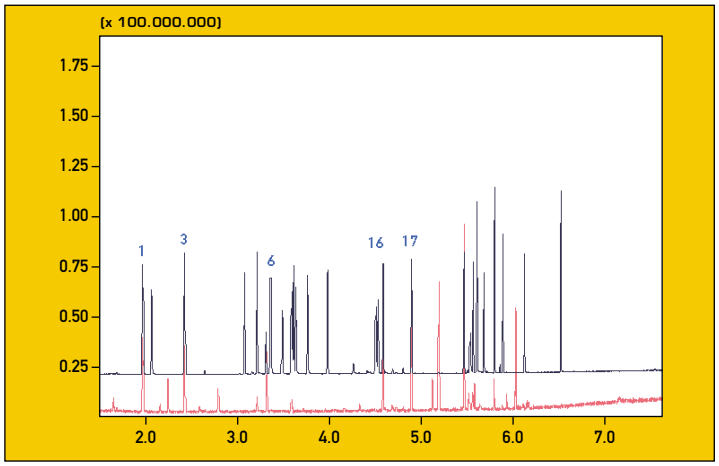

Figure 4: Peaks in the retention time segment 1 for comparison of resolution. Peak numbers are given in elutition sequence

carrier gas at the different temperatures used (linear velocity mode).

In terms of detection, the system must be able to reproduce the sharp peak shapes observed in fast GC and GCMS [5].

The peak widths in this study are about 0.5 s. Using a quadrupole GCMS system, this means a high number of scans per second for quantitative analysis which in turn needs both a high scan speed (up to 10.000 amu/s) for the mass range scanned and a small interscan deadtime. All of these requirements are met by the Shimadzu GCMS-QP2010.

Figure 2 shows the allergen standard mix measured with an SPB5 (SGE) 10 m, 0.1 mm ID, 0.1 µm. In order to compare the resolution, segment 1 has been enlarged in figure 3.

When comparing the individual peaks, it is clear that the resolution is better with the fast analysis (citral and anisyl alcohol is resolved), with also the benefit of a speed gain of a factor of 11. For the peak widths observed (FWHM approx. 0.5 s) an acquisition rate of 20 spectra per second with a mass range of 30 to 350 amu was selected. The quality of spectra obtained with the GCMS-QP2010 is very high and yield similarity indices between 94 and 98 for the allergen compounds. Linearity was tested between 4ppm and 400 ppm, and gave a regression coefficient of 0.99999 which also verifies the precision of the method.

The method was then applied to real samples. The TIC result of a perfume diluted in acetone (1:1000) and then injected is shown in figure 4 together with the standard (50-150 ppm). The allergens found in the dilution are Limonene (33.75 ppm), Linalool (27.77 ppm), Citral (Neral 12.3 ppm), Methyl Gamma Ionone (17.9 ppm), and Lilial (40.1 ppm), corresponding to a concentration 1000 times higher in the original material.

The data demonstrates the practical use of this fast method on real samples. Since the new GCMS-QP2010 can yield up to 50 spectra per second with a scan speed of up to 10.000 amu/s, the analysis can be used as a quantitative method for allergen determination.

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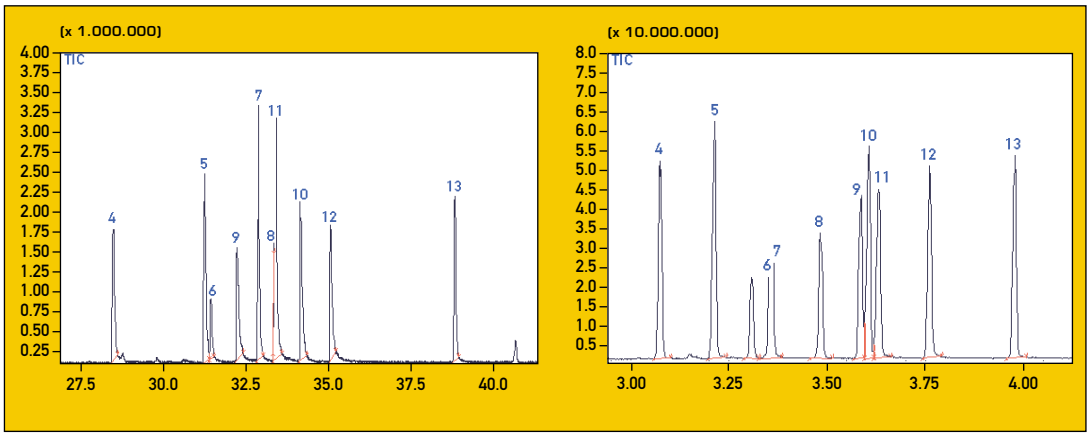

Figure 3: TICs of a perfume diluted 1000:1 in acetone and injected with the same method as figure 2. For reference, the standard is plotted above