High Speed & High Resolution GC-FID Analysis of Geranium and Limette Essential Oils

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Narrow-bore column fast GC can be considered as a routine and powerful tool that enables drastic reductions in analys times, while maintaining a high level of chromatographic resolution. With narrow bore columns the slopes of the van deemter curves (HETP= hight equivalent of a theoretical plate) beyond the HETP min are smaller compared to the one observed with standard columns. Therefore the mean linear velocity of the carrier gas can be increased to a certain extent without gradual loss of resolution. This is demonstrated in figure 1 [1].

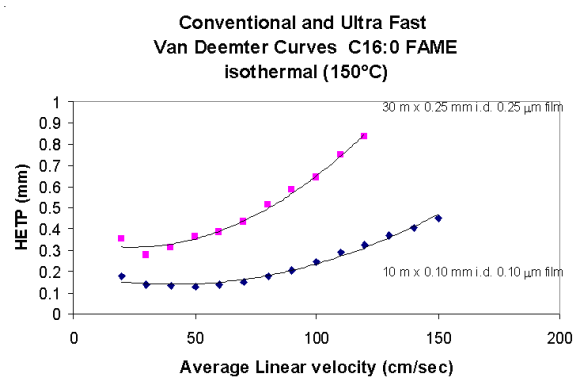


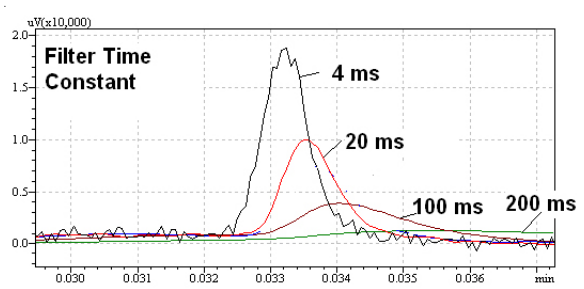
Fig. 1 : Van Deemter curves measured with C16:0 Fatty Acid Methyl Ether using a standard column (30 m, 0.25 mm, 0.25 µm) and a narrow bore column (10 m,0.1 mm, 0.1 µm)

To effectively use the resolving power of those columns the GC system has to fulfill some requirements. These are:

1. fast Sample transfer (Autosampler, liner purge) to ensure good peak shapes
2. high linear dynamic range of pressure due to the reduced inner diameter and large possible split ratios due to the reduced sample capacity for high concentration samples [1]
3. constant linear velocity mode to ensure good resolution for different temperatures (s. Fig.1)
4. High linear heating ramps as the Peaks are much sharper compared to standard GC.
5. Fast Cooling to ensure rapid cycling
6. Fast Detectors which supply
   - small Filter time constant
   - high sampling frequency

The effect of the Filter time constant is indicated in figure 2. Here chlorodecane was injected into a 1 m column (0.1 mm, 0.1 µm). To have a sharp initial bandwitdh a liner with 1 mm inner diameter was used [2].

Fig.2: Chlorodecane peak measured with different Filter time constants



The resuts obtained demonstrates that the Filter time constant τ drastically influences the peak shape of chlorodecane and for τ = 200 msec which is observed in standard GC the full width at half maximum is about 3 s. For the fast GC analysis using narrow bore columns however typically peaks with full width a half maximum of about 0.5 s are observed which needs filter time constants of about 10 ms [3].
For this experiments the filter time constant and sampling frequency were set to 4 ms and 250 Hz, respectively.
Fast GC analysis is an important tool where a rapid response is needed or for high troughput analyis like with quality control of flavours. In this work the essential oils of geranium and limette were measured using a wax column of 10 m, 0.1 mm, 0.2 µm. The results obtained are shown in figure 3. The essential oils were diluted in ethanol and the concentration was 5 % [4]. The GC program was set to 40 °C 0.5 min then 50 °C/min to 230 °C, 1 min with 60 cm/s constant linear velocity (Hydrogen). The injection volume was 1 µl with a split ratio of 400:1.
All experiments were done with the GC-2010AF equipped with AOC-20I autoinjector.

Conclusion:

Essential oil analysis with fast GC can be done with concentrations of even 5 % when high split ratios are used in order not to overload the column. No loss of chromatographic resolution was observed compared to standard-GC.

[1] L. Mondello et al: J. of chromatography A, 1035 (2004) 237-247
[2] H.-U. Baier, LCGC Europe, V16 (2003) No 12 a (Application book)
[3] J. H. Hinshaw, LCGC,V15 (2002), p152
[4] Thanks toBruno Fundenberger and the staff of Symrise France to kindly support this project

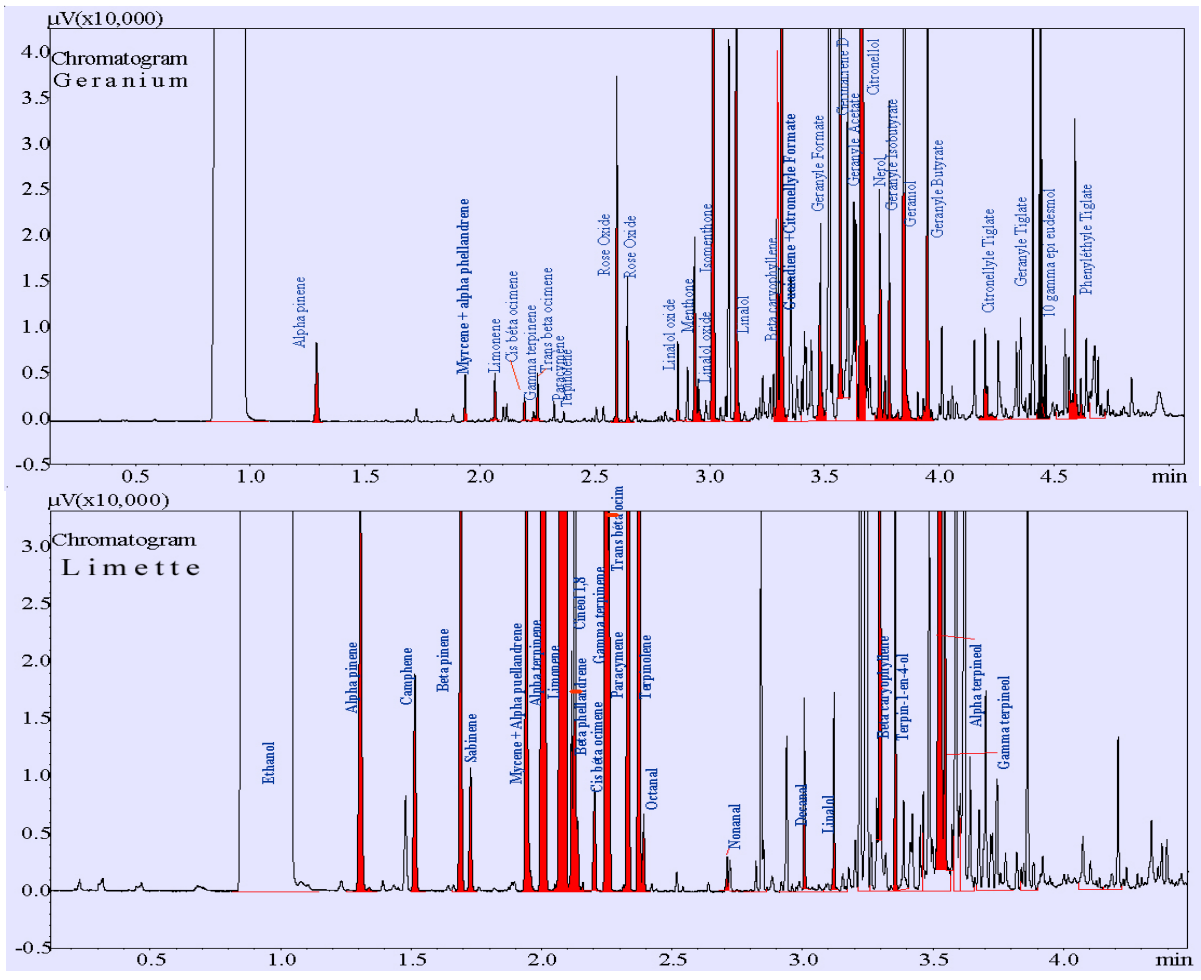


Fig.3: Chromatogramm recorded with geranium and limette essential oils [4] at a concentration of 5 % in ethanol. Injection volume: 1 µl with split ratio 400:1. Injection temperature: 250 °C. Filter time constant and sampling frequency of FID were set to 4ms and 250 Hz, respectively.