A SCENT OF FUTURE

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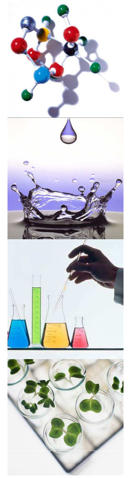

Application of the new FAST ON-COLUMN Injector*: analysis of undiluted Essential Oils with Narrow-Bore FAST-GC Columns and Conventional GC to avoid discrimination and sample alteration

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Introduction

One of the strongest limitations in the GC analysis of flavours and fragrances is the necessity to dilute the sample before the injection to make the sample amount injected compatible with the limited loading capacity of capillary columns, especially in FAST-GC. On the other hand, the introduction with vaporizing injectors affects the quality of the result due to well-known limitations such as discrimination and/or sample alteration. Moreover, high split ratio are necessary to inject undiluted samples into narrow-bore FAST-GC columns.

The new FAST On-Column inlet system is here employed that is capable to inject nanovolumes of liquid sample directly in conventional and FAST-GC narrow-bore columns.

This inlet system avoids the limitations of the hot vaporizing injectors since it enables the injection of liquid volumes in the order of nanoliters and lower, in a cool-on-column injection mode.

Automated FAST ON-COLUMN inlet system

The FAST On-Column inlet system was installed on a DANI *Master* GC. The full automation of the injection was performed through the robotic XYZ DANI *Master* AS Liquid Autosampler.

According to its original concept, this injector provides that the column enters into the needle of the syringe through a special insert liner. By capillary action, the column picks up a very small amount of sample from the needle.
The injector parts and operation steps are shown in Figures 1 and 2.

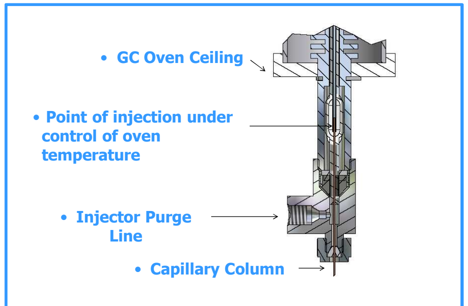


Figure 1. FAST On-Column Injector cross-section

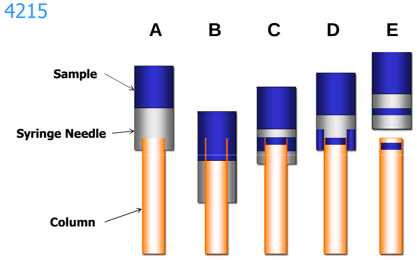


Figure 2. FAST On-Column operation steps

- *A – the syringe needle slides over the column*
- *B – the column comes in contact with the sample*
- *C, D – the liquid is sampled by capillary action*
- *E – the needle withdraws and the analysis starts*

Two main parameters are available, depth of the needle and time of insertion, to vary the injected volumes from less than 1 nanoliter up to "Large Volumes" injections.

Application

The performances of the FAST on-column injector were tested on a mandarin essential oil. Mandarin essential oil is widely used in the flavours and fragrances field. Because of the presence of important early eluting peak, its chromatographic profile well suits to this study.
A 10% oil solution in Hexane was used for standard PTV injections while a pure, undiluted essential oil was used in FAST On-Column injection with both conventional and narrow-bore columns.
The profile of the diluted essential oil injected into the PTV injector shown in the chromatogram can be compared with the profile of the undiluted essential oil injected with FAST On-Column injector. The injection of a sample volume of 0.1 – 0.2 nL with the FAST On-Column injector was obtained without any hot vaporization and split of the sample. The FAST On-Column was also applied to a conventional I.D. column by adjusting the amount injected.
No column overloading occurred even in the FAST narrow-bore columns.
No discrimination and/or alteration on the pure sample are shown on the chromatograms of both FAST and conventional separations; a slightly higher recovery of the last eluting peaks was achieved with FAST On-Column injection.

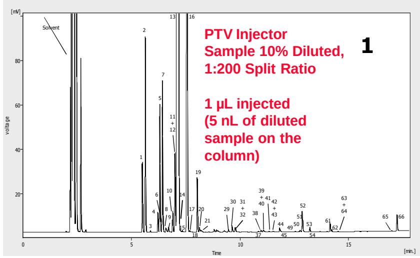
Conventional GC

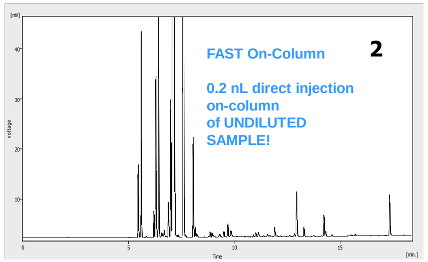
Column:
30m L x 250 µm i.d., 0.25 µm f.t. DN-5

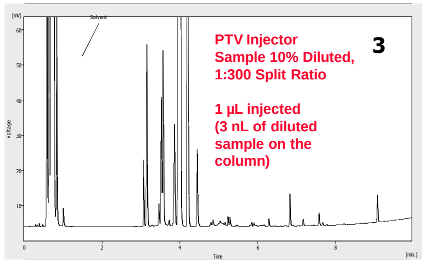
Oven:
50°C – 10°C/min – 320°C

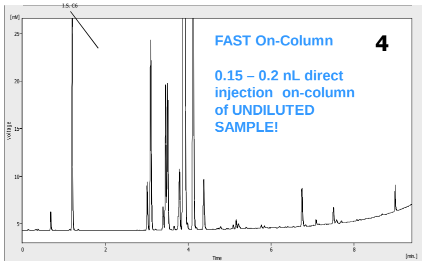
Carrier gas:
Helium @ 1.1 bar Constant Pressure

The chromatogram on the top (1) shows an injection of diluted Mandarin Oil with PTV injector (the temperature program of PTV was 50°C, 900°C/min, 320°C hold 5min). The absolute amount of pure oil entering the column was about 0.5 nL (1µL injected of a 10% solution, split ratio 1:200).
In the chromatogram below (2), the injection of a pure oil in the Cold On-Column mode is reported. About 0.2 nL of pure undiluted oil was injected. No overloading of the column occured and all the small peaks are well visible. The profile of the last part of the chromatogram, where significative peaks for the characterization of Mandarin Essential Oil (peaks 52 and 66) are present, was improved.









FAST - GC

MANDARIN OIL

Column:
5m L x 100 µm i.d., 0.2 µm f.t. DN-5

Oven:
50°C – 30°C/min – 320°C

Carrier gas:
Helium @ 3.5 bar Constant Pressure

The injection of a diluted essential oil into a FAST narrow-bore column with the PTV injector is shown in the chromatogram on the top (3). The PTV temperature program was 50°C, 900°C/min, 320°C for 5min; 1µL of a 10% solution was injected with a split ratio 1:300: the absolute amount into the column was about 3nL of diluted sample.
The injection of the undiluted essential oil into a FAST narrow-bore column is reported in the chromatogram below (4). About 0.15 – 0.2 nL liquid were directly injected into the column. No overloading occured.
To improve the detection of traces, the injected amount can be increased by increasing the residence time.

Compounds List:

1.α-thujene
2.α-pinene
3.Camphene
4.Sabinene
5.β-pinene
6.6-methyl-5-epten-2-one
7.Myrcene
8.α-phellandrene
9.Octanal
10.δ-3-carene
11.α-terpinene
12.p-cymene
13.Limonene
14.(Z)-β-Ocimene
15.(E)-β-Ocimene
16.γ-Terpinene
17.cis-sabinene hydrate
18.Octanol
19.Terpinolene
20.trans-sabinene hydrate
21.Linalool
22.Nonanal
23.p-mentha-1,3,8-triene
24.trans-pinene hydrate
25.cis-limonene oxide
26.trans-limonene oxide
27.Camphore
28.Citronellal
29.Terpinen-4-ol
30.p-cymen-8-ol
31.α-terpineol
32.Decanal
33.Citronellol
34.Nerol
35.Methyl timol
36.Neral
37.Geraniol
38.Lynalil Acetate
39.Carvone
40.2-decen-1-al
41.Geranial
42.Perylla Aldeyde
43.?
44.Thymol
45.Undecanal
46.Nonile acetate
47.Citronellyl acetate
48.Neryl Acetate
49.α-copaene
50.Neril acetate
51.β-cuebebene
52.Dodecanal
53.Methyl N-methyl anthranilate
54.β-cariophyllene
55.trans-α-bergamotene
56.α-humulene
57.2-dodecen-1-al
58.?
59.Valencene
60.α-selinene
61.α-farnesene
62.β-bisabolene
63.δ-cadinene
64.Tetradecanol
65.Tetradecanal
66.α-sinesal

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*Patent Pending