

A new Nanovolume FAST ON-COLUMN Inlet System: principle of operation, automation and applications on high temperature FAST Gas Chromatography.

Manuela Bergna (1), Stefano Galli (1)

1: DANI Instruments SpA, Viale Brianza, 87 – I 20093 Cologno Monzese (MI)

e-mail: stefano.galli@danispa.it

INTRODUCTION

A new revolutionary automated inlet system that allows injection of nanoliters of liquid directly into the column is here presented. It combines the performances of the well-known traditional On-Column Inlet with the versatility of use of a Split Injector. Particularly, it is the only injector currently able to perform a true on-column injection into the narrow bore columns used in FAST-GC, the technique more and more frequently applied because of its high resolution and short analysis time.

At present time, the existing inlets have great limitations especially if used for FAST-GC technique. In brief:

- Split Injection limitations: sample alteration and discrimination, ghost peaks and impurities coming from hot septa, very high split ratio needed for FAST-GC application (typically 1:500 split ratio or higher) which strongly affects the accuracy and repeatability of amount injected.
- PTV limitations: impurities coming from hot parts (septa), very high split ratio for FAST-GC applications, unwanted absorptions on glass wool insert.
- Traditional On-Column limitations: sample dilution is needed, Retention-Gap is necessary to prevent contamination and degradation of the analytical column, not applicable to FAST-GC.

The new FAST On-Column inlet system (*) is able to solve the listed limitations since performs an introduction of very small amount (1nL or lower) in a cool-on-column injection mode.

(*) Patent pending system by DANI Instruments SpA.

Inventor: Mario Galli, MEGA snc (Via Plinio, 29–I–20025 Legnano MI)).

Automated FAST ON-COLUMN inlet system

The FAST On-Column inlet system, installed on a DANI MasterGC, is totally automated through the robotic XYZ DANI MasterAS liquid autosampler.

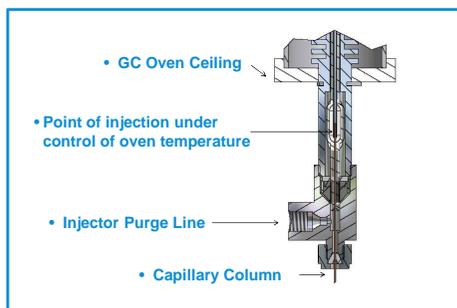


Figure 1. FAST On-Column Injector cross-section.

PRINCIPLE OF OPERATION

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 into the needle. The injector parts and operation steps are shown in Figures 1 and 2.

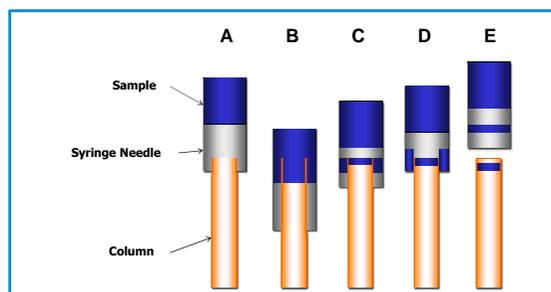


Figure 3. FAST On-Column operation steps.

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

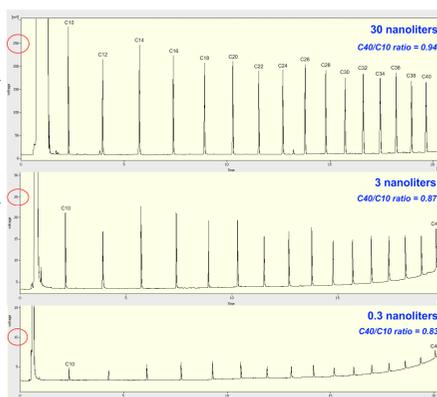
Two main variables are available, depth of the needle and time of insertion, whose variation allows to inject quantities from 0.2 nanoliter up to "Large Volume" injections.

APPLICATIONS

1. HYDROCARBONS C10 – C40

Column: 5 m L x 250 µm i.d., 0.1 µm ft. DN-1
Oven: 40°C – 15°C/min – 350°C
Carrier gas: Helium at 30 kPa

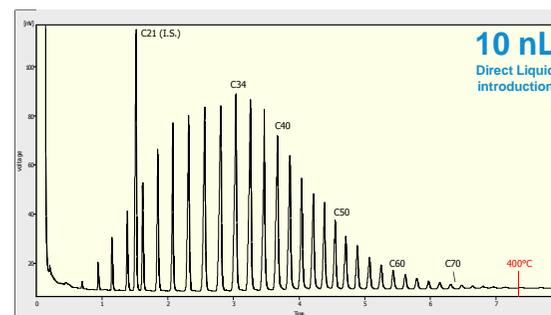
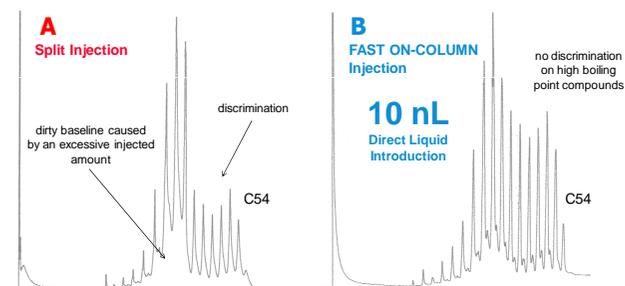
The performances of the FAST On-Column Inlet system are shown with an introduction of Alkane Mixture (FLUKA Cat.# 68281, "Alkane Standard Mixture for the assay of the system efficiency of GC's C10-C40") from 30nL down to 0.3nL direct liquid injection into the column. The discrimination is minimized as in traditional On-Column injection.



2. BUTTER TRIGLYCERIDES

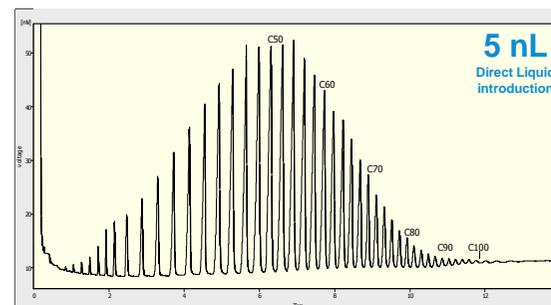
Column: 1.5 m L x 100 µm i.d., 0.1 µm ft. DN-1
Oven: 100°C – 30°C/min – 350°C
Carrier gas: Hydrogen at 150 kPa

8 min
analysis time



3. POLYWAX 500

Column: 1m L x 100µm i.d. 0.1 µm ft. DN-1
Carrier gas: Hydrogen at 200 kPa
Oven Program: 50°C – 70°C @ 140°C/min
 70°C – 115°C @ 95°C/min
 115°C – 175°C @ 70°C/min
 175°C – 300°C @ 50°C/min
 300°C – 400°C @ 35°C/min



4. POLYWAX 655

Column: 1m L x 100µm i.d. 0.1 µm ft. DN-1
Carrier gas: Hydrogen at 250 kPa
Oven Program: 50°C – 70°C @ 140°C/min
 70°C – 115°C @ 95°C/min
 115°C – 175°C @ 70°C/min
 175°C – 400°C @ 25°C/min

Thanks to the high temperature rate of MasterGC and the capabilities of FAST On-Column inlet system, very high boiling point sample have been injected directly as liquid in a narrow-bore FAST-GC column thus eliminating any sample alteration and the use of a retention gap, and performing the analysis of hydrocarbons up to C100 in less than 12 min.

