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Application of the new FAST ON-COLUMN Injector*: analysis of Explosives Mixture, according to EPA Method 8095, with FAST-GC narrow-bore dedicated columns

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Introduction

Analysis of explosives mixture is complicated by the unstable nature of the compounds themselves. Some subtances, particularly HMX, tend to decompose first in the injection port, making the conventional injection techniques incomplete for analysis of the compounds listed in the US EPA Method 8095. Moreover, the demand of a fast analysis for a rapid test of the mixtures using FAST-GC narrow bore column is increasing.
A new inlet system, called FAST On-Column, is here employed, capable to inject liquid nanovolumes directly into both narrow-bore and conventional i.d. columns. This injector avoids any alteration of the sample and makes a true on-column injection directly into FAST 100µm i.d. columns possible.
For the analytical separation, a new stationary phase, DN-TNT 8095, was used especially developed to completely resolve the 18 compounds present in the mixture in a single run. In this application, the use of a short narrow-bore column brings two main advantages: not only it reduces the analysis time without loss of resolution but also maintains the elution temperature as lower as possible, thus eliminating any thermal decomposition in the column as well.

Automated FAST ON-COLUMN inlet system

The FAST On-Column inlet system, installed on a DANI *Master* GC, is totally automated through the robotic XYZ DANI *Master* AS Liquid Autosampler.

According to its original concept, this injector allows the column to enter 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.

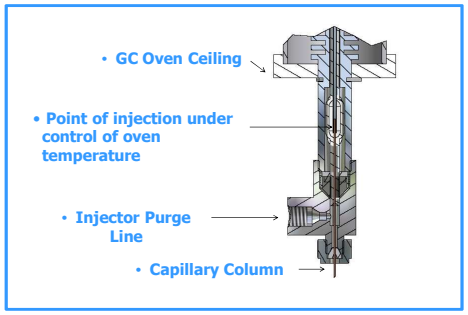


Figure 1. FAST On-Column Injector cross-section.

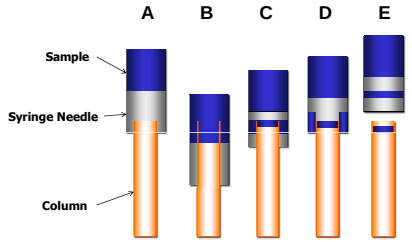


Figure 2. FAST On-Column operation steps.

- *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 variables are available, depth of the needle and time of insertion, whose variation allows to inject quantities from lower than 1 nanoliter up to "Large Volume" injections.
The new FAST On-Column inlet system avoids the limitations of hot vaporizing injectors since it performs an introduction of very small amount (1nL or lower) in a cool-on-column injection mode.

Application

A mixture of 18 compounds listed in EPA Method 8095 was analyzed. The mixture was composed by the standards listed below in 1:1:1 proportions:

•8095 Calibration Mix A (Restek cat.# 31607)
•8095 Calibration Mix B (Restek cat.# 31608)
•3,4-dinitrotoluene (ISTD, Restek cat.# 31452)

Because of the unstable nature of the compounds, a short column was used to avoid thermal decomposition in the column. In order to maintain an adequate resolution, a narrow-bore 0.1 mm column was employed. In addition, a new stationary phase (DN-TNT 8095) was developed: the selectivity was tuned to separate critical pairs of peaks on a single column. The results show a complete resolution of the 18 listed compounds. The perfect peak shape of all the components and especially of RDX and HMX demonstrates the benefits of the FAST On-Column injector, the new stationary phase and the use of short FAST-GC columns.

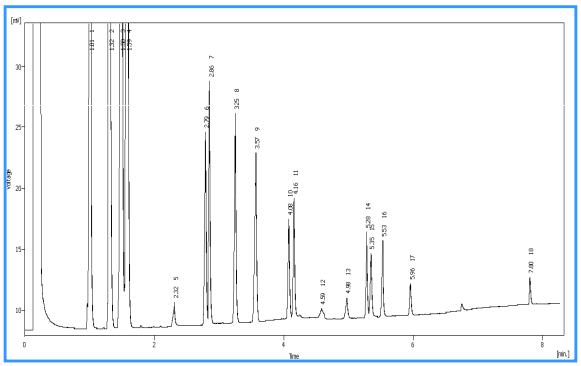
EPA 8095 Explosives Mixture

CONDITIONS

Column:
6m L x 100 µm i.d., 0.20 µm f.t. DN-TNT 8095
Oven:
90°C – 22°C/min – 280°C
Carrier gas:
Hydrogen @ 140 kPa Constant Pressure

Injector:
FAST On-Column
≈ 4nL injected directly on-column

Sample:
8095 Calibration Mix A (Restek cat.# 31607) +
8095 Calibration Mix B (Restek cat.# 31608) +
ISTD 3,4-dinitrotoluene (Restek cat.# 31452)
1:1:1 mixture



The chromatogram shows a perfect peak shape and the complete resolution of all the 18 compounds listed in the EPA method in a single analysis.
Not only the new injector and column used but also the operative conditions are important to obtain reliable results: the temperature rate was selected to obtain the shorter analysis time and the lower elution temperature.
With a 22°C/min temperature rate, the elution temperature of the last compounds, the most critical HMX, is about 260°C that is low enough to avoid thermal decomposition.

RT [min]	PEAK #	NAME	
1.01	1	Nitrobenzene	8095 Calibration Mix B (Restek cat.# 31608)
1.32	2	2-nitrotoluene	8095 Calibration Mix B (Restek cat.# 31608)
1.50	3	3-nitrotoluene	8095 Calibration Mix B (Restek cat.# 31608)
1.59	4	4-nitrotoluene	8095 Calibration Mix B (Restek cat.# 31608)
2.32	5	Nitroglycerin	8095 Calibration Mix B (Restek cat.# 31608)
2.79	6	3,4-dinitrotoluene (ISTD)	Restek cat.# 31452
2.86	7	2,6-dinitrotoluene	8095 Calibration Mix A (Restek cat.# 31607)
3.25	8	1,3-dinitrobenzene	8095 Calibration Mix A (Restek cat.# 31607)
3.57	9	2,4-dinitrotoluene	8095 Calibration Mix A (Restek cat.# 31607)
4.08	10	2,4,6-trinitrotoluene	8095 Calibration Mix A (Restek cat.# 31607)
4.16	11	1,3,5-trinitrobenzene	8095 Calibration Mix A (Restek cat.# 31607)
4.59	12	PETN	8095 Calibration Mix B (Restek cat.# 31608)
4.98	13	4-amino-2,6-dinitrotoluene	8095 Calibration Mix A (Restek cat.# 31607)
5.28	14	RDX	8095 Calibration Mix A (Restek cat.# 31607)
5.35	15	3,5-dinitroaniline	8095 Calibration Mix B (Restek cat.# 31608)
5.53	16	2-amino-4,6-dinitrotoluene	8095 Calibration Mix A (Restek cat.# 31607)
5.96	17	Tetryl	8095 Calibration Mix A (Restek cat.# 31607)
7.80	18	HMX	8095 Calibration Mix A (Restek cat.# 31607)

Table 1. Peak identification.

Conclusions

The new DANI FAST On-Column is the only injector capable to inject very small volumes of liquid sample directly into FAST narrow-bore capillary columns: the minimum quantities, in the order of nanoliters, is compatible with the reduced capacity of the fast colums.
This avoids the use of hot split injector thus eliminating any risk of thermal decomposition of the components during vaporization. The analysis of explosives, well-known to be very sensitive to thermal degradation, take several advantages from the contemporary use of a cold injection and a short narrow-bore FAST column: thermal decomposition occurs neither in the injector nor in the column and the separation is obtained in a shorter analysis time. Moreover, a new stationary phase DN-TNT 8095 with a tuned selectivity was developed that enables the complete resolution of all the components in less than 8 minutes.

*** Patent Pending**