NEW GC / TWO-STAGE DIFFERENTIAL MOBILITY ANALYSER FOR THE SCREENING OF EXPLOSIVES IN CARGO

Gonzalo Fernández de la Mora,[1] Mario Amo González[1], Juan Fernández de la Mora[2]

[1]SEADM S. L., Boecillo, Spain; [2]Yale University, Mech. Eng. Dept., New Haven, CT 06520-8286, USA

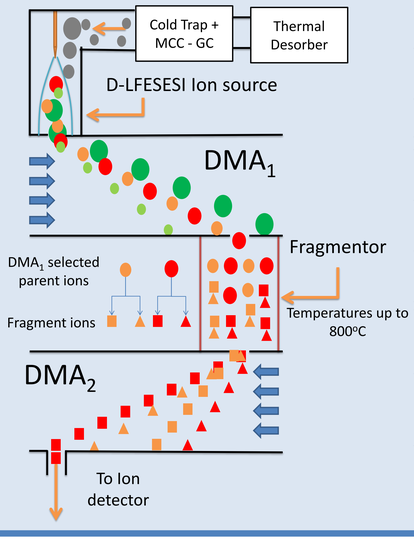
Abstract

The capability to detect explosive vapours in the atmosphere is a long sought goal in the security area. Present state of the art is centered in two solutions: IMS, which is only practical for very volatile explosives (EGDN), and mass spectrometry, which is expensive and bulky. Our aim is to detect explosive vapours in the atmosphere through the coupling of a fast GC column with two Ion Mobility devices, in order to obtain a low cost, yet sensitive and discriminating, detector.

Two differential mobility analyzers (DMAs) acting as narrow band mobility filters are coupled in series, with a thermal fragmentation cell placed in between, such that parent ions selected in DMA_1 are fragmented in the cell at atmospheric pressure, and their product ions are analyzed on DMA_2.[1] A key feature of the tandem DMA is the short residence time (~0.2 ms) of ions in the analyzer, compared to tens of milliseconds in drift tube ion mobility spectrometers (IMS). Ion fragmentation within the analyzer and associated mobility tails are therefore negligible for a DMA but not necessarily so in conventional IMS.

The presented technology has demonstrated Limits of Detection in the order of 1 pg for TNT for atmospheric samples of 500 L of air.

Methods



Blank Atmospheric Samples: comprised of 500 L of air sampled in Boecillo at the end of July, when de vapor concentration is maximum according to a previous study.[2]

Filters: Fiber glass/ stainless steel coated with Tenax GR. Allows sampling flow rates in the range of 100 – 1000 L/min.

Thermal Desorber: The filter is inserted in the desorber and desorbed at a flow rate of 0.2 L/min and a fixed temperature of 200 °C.

Cold Trap: The vapors liberated in the desorber are condensed and retained in a cold trap at 0ºC. The cold traf is built from a silica lined stainless steel tube 1/18".

Multicapillary Column GC (MCC-GC): 20 cm length, 1000 capillaries in parallel, 40 µm capillary diameter, 0.2 m of OV-5 Stationary liquid phase.

DMAs: Low residence time (200 µs), high transmission (~50 %), high resolution (up to 110) [3]. The DMA_1 selects the explosive parent ion which enters the fragmentor, whereas the DMA_2 clasifies the fragment ion generated.

Fragmentor: built from metal and ceramics is capable to reach temperatures up to 800 °C in order to break the more resilient species. The ion transmission inside the fragmentor takes place by electric fields, minimizing ion looses against the walls.

Ion Detector: For the time being a Mass Spectrometer working in single quadrupole mode is being used. However the m/z separation is not being used, representing the Total Ion Current (TIC). Once fixed the configuration and optimized the parameters the MS will be replaced by an electrometer.

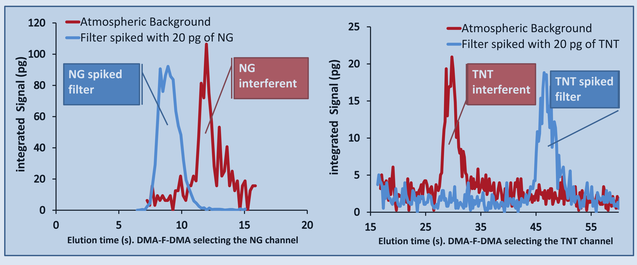
Results

The atmospheric background was evaluated for the following explosives: EGDN, NG, PETN, TNT and RDX. The analysis results are shown in Table 1.

Configuration 3: MCC-GC DMA-F-DMA									
Expl.	Parent Ion	Fragment. Temp. (ºC)	Product Ion	Analysis interval (s)	GC Temperature (ºC)	GC integration interval (s)	Integration time (s)	Gain (counts/pg)	Atmospheric Background (pg)
EGDN	$[M+Cl]^-$	145	NO_3^-	0-6.5	110	2.7-3.9	1.2	133	22
NG	$[M+Cl]^-$	145	NO_3^-	6.5-16	110	8.5-9.7	1.2	1136	7.0
TNT	$[M-H]^-$	400	$[M-H]^-$	16-63	110	46.7-48.7	2.0	24079	2.4
PETN	$[M+Cl]^-$	189	NO_3^-	63-102	110	86-89	3.0	1126	6.3
RDX	$[M+Cl]^-$	280	NO_2^-	102-180	110	118-121	3.0	215	89

Table 1. Atmospheric background and gain for the explosives, and analysis and post-processing parameters. Both gain and background are calculated during the integration interval.

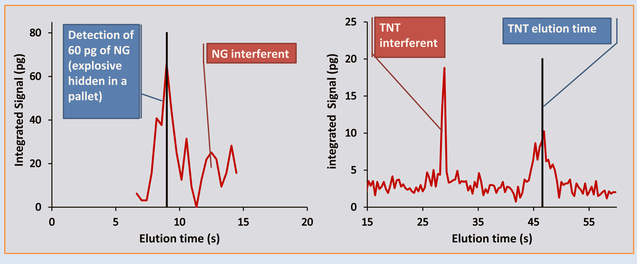
Blank and loaded samples



Graph 1. **Left.** Blue: Elution of a pipette of 100 pg of NG overlapped with the analysis of an atmospheric sample containing 500 L of air (red). The DMAs and the fragmentor are selecting the NG channel.

Right. Elution of a pipette of 20 pg of TNT overlapped with the analysis of an atmospheric sample containing 500 L of air (red). The DMAs and the fragmentor are selecting the TNT channel.

Explosive Hidden in Pallets



Graph 2. **Left.** Detection of 60 pg of NG from an air sample from a loaded pallet. The pallet is built form 9 cardboard boxes of 40 x 40 cm. The explosive (~ 20 g) is inside the central box.

Right. Detection of 10 pg of TNT from an air sample from a loaded pallet. The pallet is built form 9 cardboard boxes of 40 x 40 cm. The explosive (~ 20 g) is inside the central box, which has some small holes.

Conclusions

- MCCGC-DMA-F-DMA technology has demonstrated atmospheric backgrounds in the order of 1 pg in samples of 500 L of air taken during the hotter days of summer (high content of nitrated interferents).
- The performance of the GC system is still far from its optimum, so the room for improvement is remarkable.
- The analysis time takes 3 minutes and a half in the current configuration (1 minute for the desorption and trapping + 2.5 minutes for the analysis). After optimizing the GC performance, this analysis time can be further improved.

References

[1] M. Amo-González, I. Carnicero, S.Pérez, R. Delgado, G. A. Eiceman, G. Fernández de la Mora, J. Fernández de la Mora, *Anal. Chem.* 90 (11), 6885-6892 (2018).
[2] Zamora, D.; Amo-Gonzalez, M.; Lanza, M.; Fernandez De La Mora, G.; Fernandez de la Mora, J. *Anal. Chem.* 90(4), 2468–2474 (2018).
[3] Amo-González, M.; Pérez, S. Planar Differential Mobility Analyzer (DMA) with Resolving Power of 110. Anal. Chem.. 2018

Spectrometry for Security Applications - First International Workshop FH Vorarlberg, Dornbirn, Austria, 10th – 13th February 2019