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# ABSTRACT

In this paper the authors will present a new device, to detect explosive, as a whole system. The physical fundamentals are discussed as well al the break down of the system. The device is able to 'sniff' the components of the explosive following recognition of the molecular compounds. The design process and performances, especially resolution and detection limits, will be addressed, as well. To finalize two application scenarios will be presented IONER CHECKPOINT, for parcel bombs, concealed IED in briefcase or similar and CHECKPOINT for screening people at the entry gate. Next developments as a mobile IED finder and ultra high resolution DMA will be described.

# **1.0 INTRODUCTION**

Among existing technologies, a well established one is the Ion Mobility Spectroscopy (IMS). This technique has probably been more widely developed for commercial applications of trace explosives detection than any other. There are a number of features that make IMS attractive for the trace detection of explosives: response time of only a few seconds, proven ability to detect a number of key explosives and sensitivity in the part per billion level among others. Main drawback of these detectors is their low resolving power, usually bellow 30 [1].

The main problem with explosives is that they are frequently present at extremely low levels and may be in complex mixtures containing significant amounts of contaminants. Therefore there is a great interest in more sensitive instrumentation and improved methods for sample separation, concentration and cleanup.

The new generation of analyzers based on ion detection could include:

- improve detection limits by limiting fragmentation of ions;
- improve selectivity through the addition of a reliable and fast GC inlet or another separation method;
- improve speed and economy through small, mass produced analyzers;
- improve deployability through the use of a non-radioactive source;
- improve interlaboratory comparisons through standardized libraries; and
- enhance instrument performance through advances in sampling.

RAMEM has intended to address most of the above requirements with the IONER new explosive detection system.

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# 2.0 SYSTEM ELEMENTS

The detector system is made by several modules serially connected (fig. 1). Each module is designed to add discrimination capacity to the system. Samples are first concentrated in the concentrator and then ionized in the ionizer. After the ionizer the injector focuses the ionized compounds into the DMA when they are classified and detected. Optionally there is an interface with a TOF to further increase the resolving power of the system. All the modules are controlled by an appropriate hardware and software. An international patent application covering the whole system has been filed.



Figure 1: System schematics

#### 2.1 Concentrator

The concentrator is designed to take high volume of sample, concentrate it and deliver to the ionizer module. Conceptually is composed of two transverse circuits that cross in a trap filled with and adsorbent. The main line carries high flows from outside and the secondary line carries the concentrated sample to the ionizer. The adsorbent is chosen in a way to provide selectivity to the instrument. In normal mode of operation the concentrator has two cycles: adsorption cycle at low temperature and desorption cycle at high temperature. To ensure a continuous operation at least two lines with two traps are necessary. When one of the lines is in adsorption mode the other is in desorption mode. The concept can be easily extended to more lines, so it is possible to have multiple traps with different adsorbent materials selective to a specific compound.

A simplified diagram of the concentrator is shown in figure 2. This design has two lines with two traps A and B. In this figure trap A is in desorption mode and trap B is in adsorption mode. The module is designed to admit a flow up to 100 L/min. Maximum desorption flow is 5 L/min. A concentration factor of 100 is expected.





Figure 2: Concentrator schematics

#### 2.2 Ionizer

Several ionization methods can be used in the equipment: electrospray, corona charging, chemical ionization and radioactive ionization. The preferred one is chemical ionization both for high efficiency and selectivity. One of the systems that employ a selective volatile ionization is chemical ionization by proton transfer reactions [2]. A reaction is established between a secondary protonated compound and the target molecule. In the case of water as the secondary compound the reaction is:

 $M + H_3O^+ \rightarrow MH^+ + H_2O$ 

where M is the target compound. Only the molecules with a proton affinity greater than water are ionized. Different groups of target compounds can be ionized with different secondary compounds. Apart from water, ammonia and methane are commonly used.

We have design a proton transfer ionizer in which water molecules are ionized and then transferred to a mixing chamber where the proton transfer reaction with the sample takes place.

# 2.3 Injector

The injector is the interface between the ionizer and the DMA. Basically is a cavity where the ions and the carrier gas are separated due to the combined action of electrostatic lens and the aerodynamic field. The cavity has one inlet for the carrier gas with sample and two outlets, one for he carrier gas and other for the ions. The second one is a narrow slit that connects directly with the DMA. When the DMA is operated at high Reynolds numbers the slit is blocked for the passage of air and only ions with a defined range of electrical mobility are allowed to enter the DMA. The fact that there is no interchange with the DMA sheath flow results in a resolution improvement. Figure 3 shows the interface between the injector and the top of the DMA with the velocity fields contours.





Figure 3. Computer simulation of ions trajectories inside the injector. Left ions velocity contour (coloured), air trajectories (grey). Right: ions trajectories (grey), air velocity contour (coloured)

# 2.4 DMA

RAMEM is the pioneer company in the developments of flat DMAs. The flat DMA is a variation of cylindrical DMAs that shows many advantages with respect to them. On one hand, the plane DMA is significantly smaller and lighter than the original one. Moreover, the manufacture of the plane DMA is less costly and it is also more user friendly, i.e. easier to assemble and handle, than the cylindrical one. With a flat design it is possible to obtain higher mechanical precision than with the cylindrical ones. Other favourable functional aspect is that ion losses in the inlet region are much reduced in the plane DMA, as compared to the cylindrical one.

Basically it consists in a main circuit in with a sheath gas close to atmospheric pressure is recirculated. The core of the DMA is a narrow section where ions are classified (classification area). In this region a transversal electric field is established by applying a voltage between two opposite walls. The ions injected parallel to this field will move under the influence of these two fields and their trajectories will be dependent on their electrical mobility Z:

# $Z = \frac{\vec{u}}{\vec{E}}$

where u is the drift velocity and E is the electric field. Only ions within a range of electrical mobilities are collected at the output slit and measured by means of an electrometer. The DMA principle of operation is shown in fig. 4.





Figure 4. DMA principle of operation

A whole spectrum of mobilities is obtained making a voltage scan. Each peak of the spectrum corresponds to a defined mobility. Usually the resolution of the analyzer is expressed as resolving power:

 $R = \Delta Z/Z$ 

Where Z is the peak mobility and  $\Delta Z$  is the full with at half height of the peak. The resolving power for an ideal flat DMA when Brownian diffusion is taken into account is proportional to the square root of the Peclet number and thus of the Reynolds number [3]. Turbulence produces degradation of the resolving power. In order to assure high Reynolds numbers with low turbulence it is necessary to include some elements for flow conditioning. In the rest of the main circuit a series of elbows, grids and other flow conditioning elements are placed. Intensive computer fluid dynamics simulations (similar to those of the aero dynamic tunnels) have been made to minimize the turbulence in the classification area. As an example figures 5 and 6 show simulated air streamlines around an elbow and a grid.



Figure 5. Computer fluid dynamic simulations of air streamlines around an elbow.





Figure 6. Computer fluid dynamic simulations of air streamlines around a grid.

In house designed turbo pump provides the main line with a very stable clean sheath flow. This pump operates over 50000 r.p.m. and is controlled in a closed loop way so that flow precision better than 0.1% can be achieved.

With all these improvements resolving powers higher than 130 are expected.

#### 2.5 Sensor array

In the previous paragraph we have mentioned the necessity of performing a voltage scan to obtain a complete mobility spectrum. This can be time consuming. An alternative way of obtaining the mobility spectrum in a fraction of that time is to place a sensor array instead of or in combination with the outlet slit. The sensor array is formed by a series of conducting lines separated by insulator, each line being connected to a low noise (< 0.5 fA p-p) in house designed electrometer. We have used platinum deposited onto alumina substrate by sputtering to guarantee good isolation between lines. A detail of the deposition is shown on the confocal micrograph of figure 7.

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Figure 7. Confocal micrograph of a reduced area of the sensor array

# 2.6 TOF interface

When the DMA resolving power is not enough a mass spectrometer can be used for detection at the outlet of the DMA. An interface has been designed to connect the DMA to a time of flight mass spectrometer (TOF-MS). The interface enclosed in a sealed metal cylinder connected to a vacuum pump. Simultaneous application of rf and dc fields produce ion focussing in a low pressure (around 10 mbar) atmosphere. Transmission efficiencies over 90 % are expected. Amplitude of the rf field is about 300  $V_{pp}$  at frequencies up to 2 MHz. The dc potentials applied are of the order of 50 V. Figure 8 shows a view of the TOF interface



Figure 8. 3-D view of the TOF interface



#### **2.7 Control module**

To assure the correct operation of the different modules and perform automated measurements a control module has been developed. It is based in a central processor that communicates with the electronic interfaces through a digital series protocol (RS-485 or MODBUS). This digital bus is connected to an embedded computer that can be accessed locally through a touch panel or remotely through an Ethernet bus. Figure 9 shows the graphic user interface.



Figure 9. Graphic user interface

# 2.8 Overall system performance

The overall expected system performance is shown in table 1.

Table	1.	System	performance
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Parameter	Value
Resolving power	> 130
Detection limit	< 1 ppt
Response time	< 30 s
Selectivity	< 1% false positive/negative rate



# **3.0 EXPERIMENTAL**

DMA body has been calibrated with an electrospray source. Test compound used has been a solution of tetra heptyl ammonium bromide (THAB) in ethanol at a concentration of 10  $\mu$ g/L. The spectrum of this compound at a flow sheath of 90 L/min is shown in figure 10. By varying the sheath flow the resolving power can be obtained as a function of the DMA classification voltage (figure 11).



Figure 10. Mobility spectrum of THAB





Figure 11. Experimental and theoretical resolving power of DMA for THAB

The DMA has been tested also with a explosive and a simulant of a chemical warfare agent. Fig. 12 shows the mobility spectrum of a pentaerythritol tetranitrate (PETN) solution of 1 ppmv in ethanol obtained with electrospray. Fig. 13 shows the spectrum of a solution of 1 ppmv of the simulant dimethyl methyl phosponate (DMMP) obtained with electrospray too. In both cases there is good agreement between measured mobilities and those found in literature [4, 5].





Figure 12. Mobility spectrum of a solution of 1 ppmv of PETN in ethanol



Figure 13. Mobility spectrum of DMMP



# 4.0 SCENARIOS

Two scenarios have been devised based on NATO NIAG SG-84 study, one checkpoint for people and other for parcels, briefcase and hand-baggage. As with the other modules computer fluid dynamic simulations have been made. Fig. 14 shows one of these simulations for the parcel scenario. Figure 15 shows a photograph of the checkpoint prototype.



Figure 14. CFD simulation of a chekpoint for the parcel scenario



Figure 15. Photograph of the checkpoint prototype

# **5.0 ONGOING WORK**

#### 5.1 Source finder

We have developed a robotised odour tracking system able to find autonomously the source of volatiles compound. The sensor which detects volatile substances leads the robot to seek after the source with a gradient based algorithm. This prototype would be the basis for a more advanced robot that is being developed by RAMEM that uses other more efficient searching strategies.



#### 5.2 Very high resolution DMA

For filed applications requiring high resolving power it would be advantageous to replace the mass spectrometer due to difficulty of handling. We are working in a non conventional DMA design which will reach a resolving power of 2000 without the need oh high Reynolds numbers or high voltages. An international patent application has been already filed.

#### 6.0 REFERENCES

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