

# Ceramic DMS—type detector

M. Jakubowska · M. Maziejuk · M. Ceremuga ·  
J. Siczek · W. Gallewicz

Received: 2 February 2012 / Revised: 22 March 2012 / Accepted: 26 March 2012 / Published online: 8 May 2012  
© The Author(s) 2012. This article is published with open access at Springerlink.com

## Introduction

DMS Technology is based on ion segregation phenomenon when flowing through the volume of the detector. DMS detector is made up with two parallel ceramic plates with electrodes layered on each plate. High voltage and high frequency of electric field generated in detector's volume causes ion segregation on collection electrode (see Fig. 1).

Observed ion segregation in flowing gas is caused by different mobilities of the analyzed chemicals in fields characterized by lower and higher intensity. Ion mobility value is dependent on ion mass, charge and velocity of flowing gas [1–4].

Figure 2 depicts example flow gas through detector's volume. Variable electric field put on electrodes causes capturing of these ions, which mobility does not enable stable (parallel to detector's plates) flow through detector's

---

**Objective scope** Despite of extreme progress in IMS technology detection CW agents is still serial problem. In fact detection CW agents generally is not a problem. Problem is to detect very low concentrations with low false alarms level. It is still challenge for R&D teams. Target of this work is elaboration and investigation DMS type detector fulfilling above needs.

---

M. Jakubowska  
Warsaw University of Technology,  
Warsaw, Poland

M. Maziejuk (✉) · M. Ceremuga · J. Siczek  
Military Institute of Chemistry and Radiometry,  
Al. Gen. A. Chruściela 105,  
Warsaw, Poland  
e-mail: m.maziejuk@wichir.waw.pl

W. Gallewicz  
Galwes Company,  
Warsaw, Poland

volume. Thus, phenomenon of ion mobility of ions flowing through the detector variable on compensated electrical field, makes detector as special ion filter.

For DMS detector, ion mobility is defined by following equation:

$$K\left(\frac{E}{N}\right) = K(0) \left\{ 1 + \alpha \left(\frac{E}{N}\right) \right\} \quad (1)$$

In low intensity electrical fields,  $\alpha$  equals 0.

In cases, where intensity augments, thus effective temperature of ion raises, and in that case it's temperature is higher than ambient temperature [1].

Important property determining low field condition is not just the field but the field normalized to the drift gas molecular density,  $E/N$ , the unit being the Townsend (one Td  $1 \cdot 10^{17} \text{ Vcm}^2$ ). Ions in fields below 10 Td have  $T_{\text{eff}}$  essentially equal to  $T$ , the temperature of the surrounding gas [4–7].

The reactions of ions in ion mobility spectrometers under low field conditions at atmospheric pressure and well-defined temperatures can therefore provide kinetic and thermodynamic data [see 8–15].

Article describes construction technology of DMS detector, and preliminary tests of chemical warfare agents (CWA) detection using above detector.

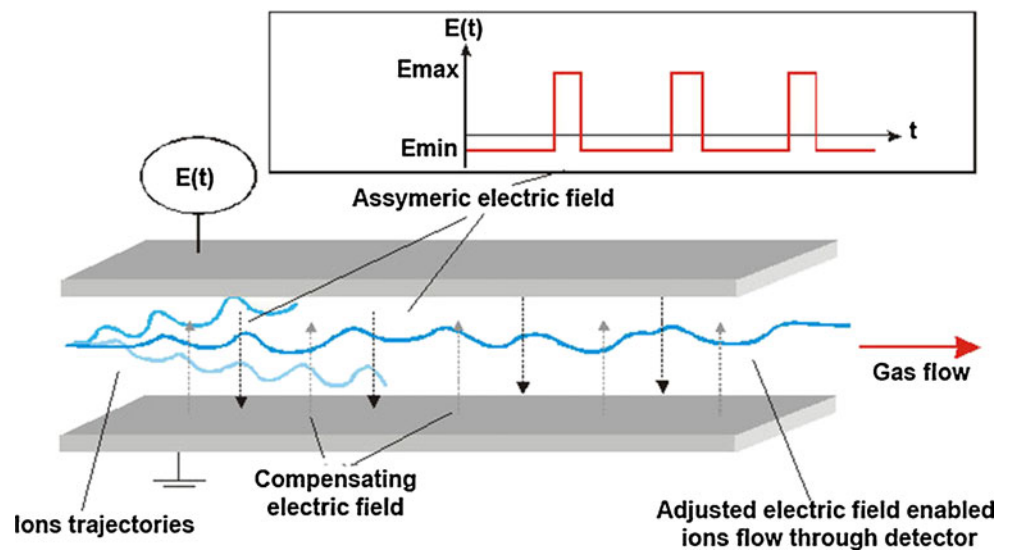
## Construction of ceramic DMS chamber

Schematic cross section - see Fig. 3

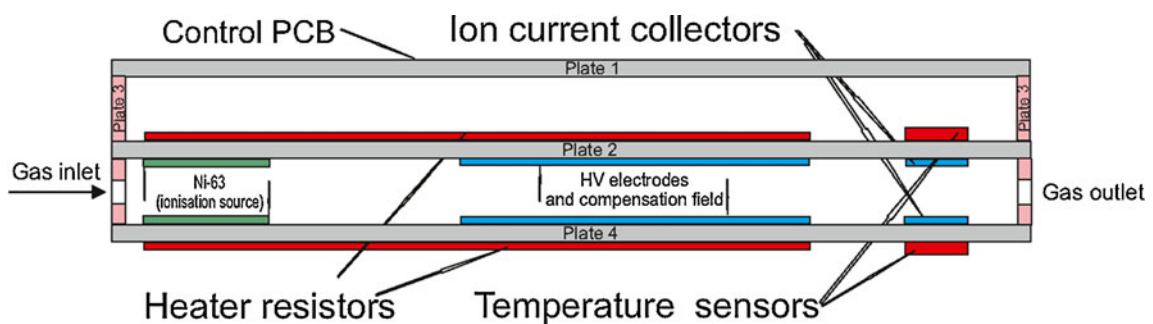
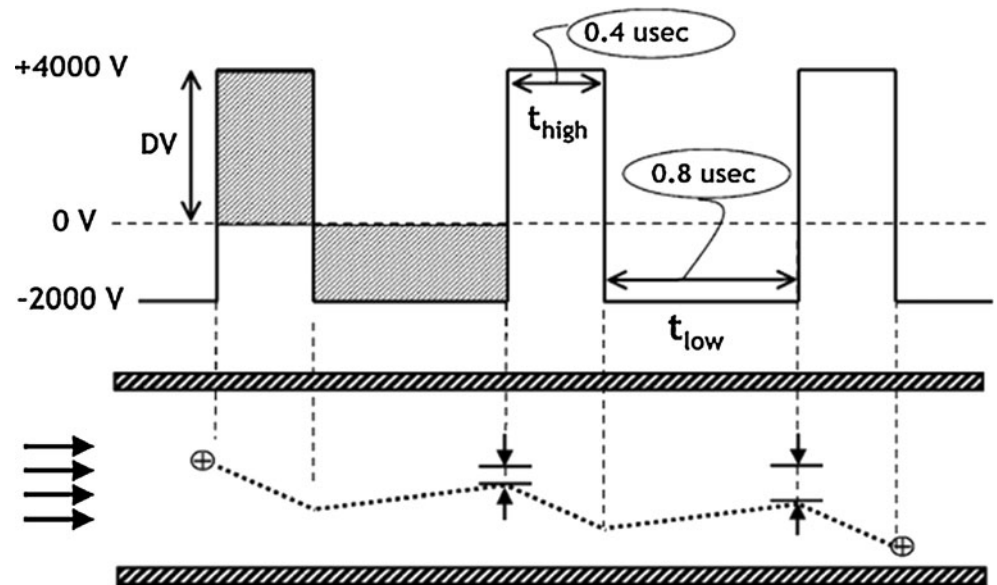
Plate no. 1 with ion current amplifier, ADC, and system control;

Plates no. 2 and 4 (ceramic):

**Fig. 1** Diagram of DMS detector composition.



**Fig. 2** Diagram of the ion flow through detector's volume.



**Fig. 3** Scheme of DMS chamber design

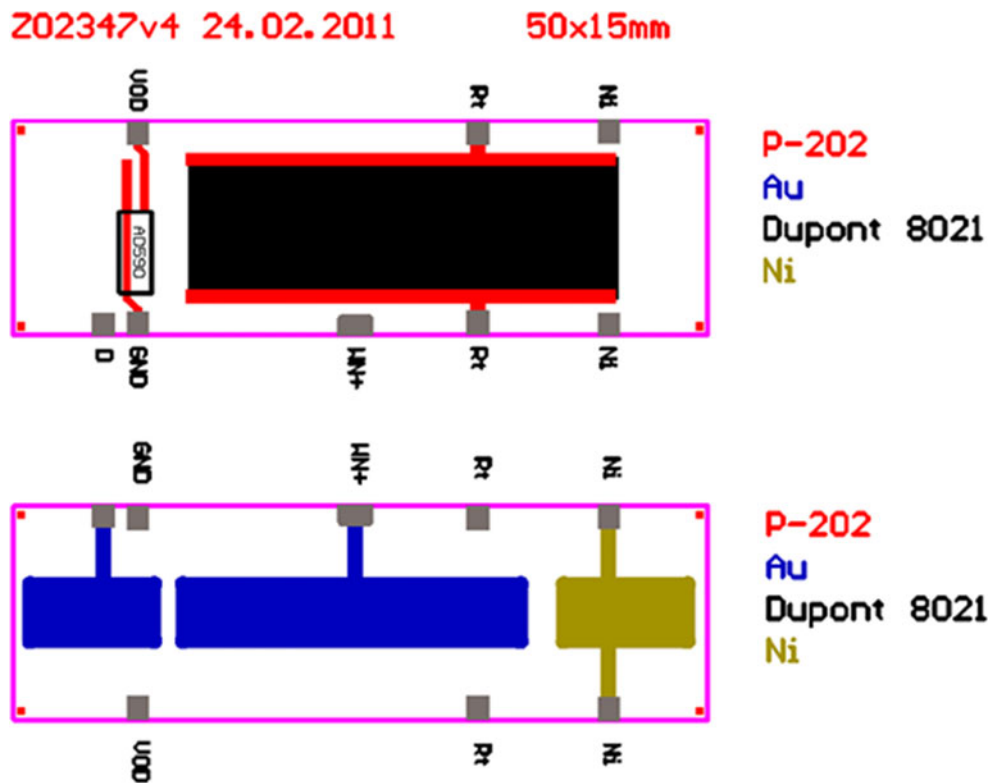


Fig. 4 Outer and inner (from top) surfaces of plate number 2 and 4

- outer surface with enclosed heater resistors and temperature sensors;
- inner surface with enclosed chamber electrodes and air ionization system;

Plate no. 3 - ceramic plate with edge connectors.

Explanation to Fig. 4:

- Heater resistors ( $\text{RuO}_2$ ) on both pictures between **Pt-Pt** connectors
- Temperature sensor AD590 on upper picture between **VOD-GND** connectors
- Ionization source Ni-63 on lower picture between **Ni-Ni** connectors

- High Voltage on lower picture **WN+**connector
- Ion current collector on upper picture **D** connector

To manufacture this chamber planar thick-film technology was used. On isolated substrate one by one conductive paste, isolation material and resistive material was screen-printed. Noble metals pastes used as electrodes and detection surfaces protect equipment from corrosion and guarantee long and stable work without changing parameters.

Chamber consist of four ceramic plates made from alumina 96 %  $\text{Al}_2\text{O}_3$ . Two main plates (number 2 and 4) are made from ceramic thickness 1/40". On this substrates with the use of screen-printing techniques the following layers were placed and fired:

1. Conductive layer made of Ag-Pd paste for conductive contacts and power points for heater resistor;
2. Conductive layer made of Ag paste for edge connectors;
3. Conductive layer made of Au paste for signal and detection electrodes;
4. Resistive layer made of  $\text{RuO}_2$  paste;
5. Conductive layer made of Ni paste followed by nickel plated electrode as basis to cover with Ni-63 - ionization source for gas flowing over electrode;

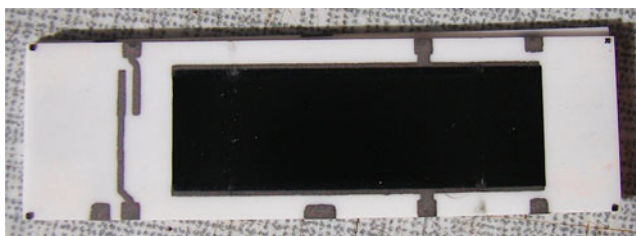
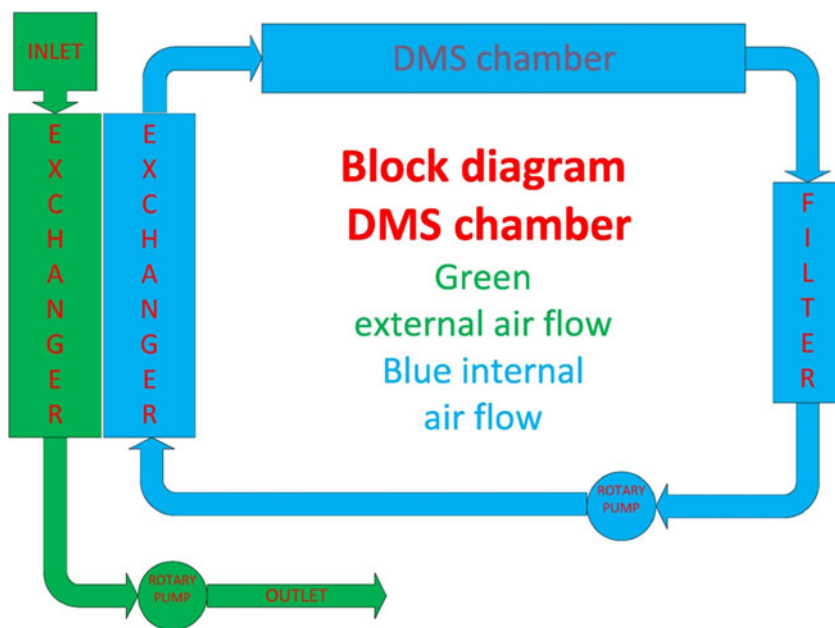


Fig. 5 Plates number 2 and 4 stucked together

**Fig. 6** Block diagram of “closed” gas exchange system used in DMS chamber



6. Low temperature melting glaze layer as welding element joining all layers (Fig. 5).

Ni electrode placed below flowing gas covered with radioactive Ni<sup>63</sup> makes gas ionized. Ions are separated in strong asymmetric electric field.

Ion current value from ten to twenty picoampers is received by detecting electrodes, separately for positive and negative ions and amplified in next layer in chamber (plate No 1).

It serves to dose carrier gas into spectrometer. We have used a purified air with relative humidity not exceed than 5 % as carrier gas.

Exchanger consists of two air chambers divided by semi-permeable membrane. Two streams of air are flowing along the membrane in different directions. One stream is carrier gas second – atmospheric analyzed air.

Vapours of chemical compounds are going through membrane and mix with carrier gas. Then they are analyzed in spectrometer and in the end settle in filter.

Heater resistors has been mounted on exchanger body (black surface on Fig. 8).

**Investigation of ceramic DMS chamber**

DMS chamber manufactured as described above was tested in two gas flow systems:

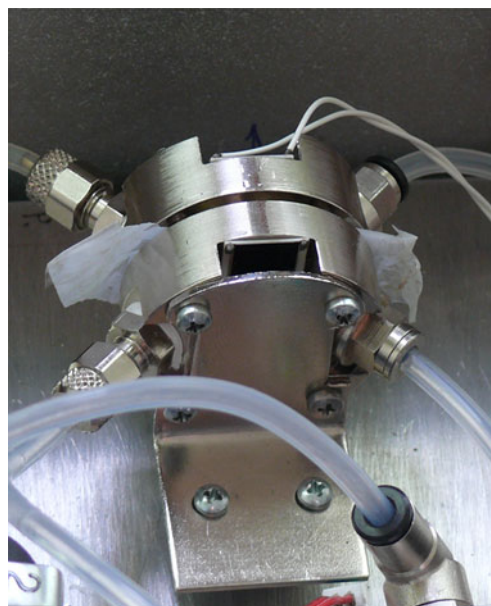
- “Closed” with semi-permeable membrane
- “Open” with direct gas intake into chamber

Gas flow systems used during our investigation is presented in Figs. 6 and 7.

The most important part in closed gas flow system is gas exchanger module. See Fig. 8.



**Fig. 7** Block diagram of “open” gas exchange system used in DMS chamber



**Fig. 8** Gas exchanger module

It speed up mixing analyzed substances with carrier gas and allow analyzer quickly clean up after finish the measurement.

### Results of investigation DMS spectrometer model with open and close gas flow system

During project realization some pilot survey was investigated. Target of this survey was:

- optimization of construction of electronic circuits,
- optimization of construction of pneumatic system,
- testing properties semi-permeable membrane.

Investigation was done for several CW agents with concentration above  $10 \mu\text{g}/\text{m}^3$ . For all investigated CW agents specific reactions were obtained and identification of all these compounds was reached.

Oscillograms from DMS spectrometer with closed gas system are presented on Figs. 9, 10, 11, and 12.

For positive ions some small peaks appears from unknown compounds. They are not valuable and have no meaning on CWA detection. Reactant ion peak (RIP) occurs

for both positive and negative ions. See Fig. 9 reference spectrum.

On Fig. 10 for big intensity peak (monomer and dimer) for positive ions is showed. Sophisticated mathematical methods allow extract dimer from this big one peak. For calculations coefficient  $\alpha$  was defined only for monomer.

For negative ions we can observe two small peaks. These are probably residuals from GA (tabun) in analyzed air. See Fig. 10.

For Vx detection picture show positive ions peak with difficult to differentiate monomer and dimer. For negative ions practical no detection (Fig. 11).

For mechlorethamine (HN-3) reaction is observed for both positive and negative ions (one peak for each polarization). For  $\alpha$  coefficient calculations only negative ions results were taken.

Two oscillograms from DMS spectrometer with open gas system are presented on Figs. 13 and 14.

For open gas system some new peaks occurs from unknown compounds. These are probably VOC's (Volatile Organic Compounds) which pops up for compensation voltage suitable for Chemical Warfare Agent detection. See Fig. 13.

For open gas system appears stronger detection signal. For GB (sarin) concentration near  $20 \mu\text{g}/\text{m}^3$  signal has

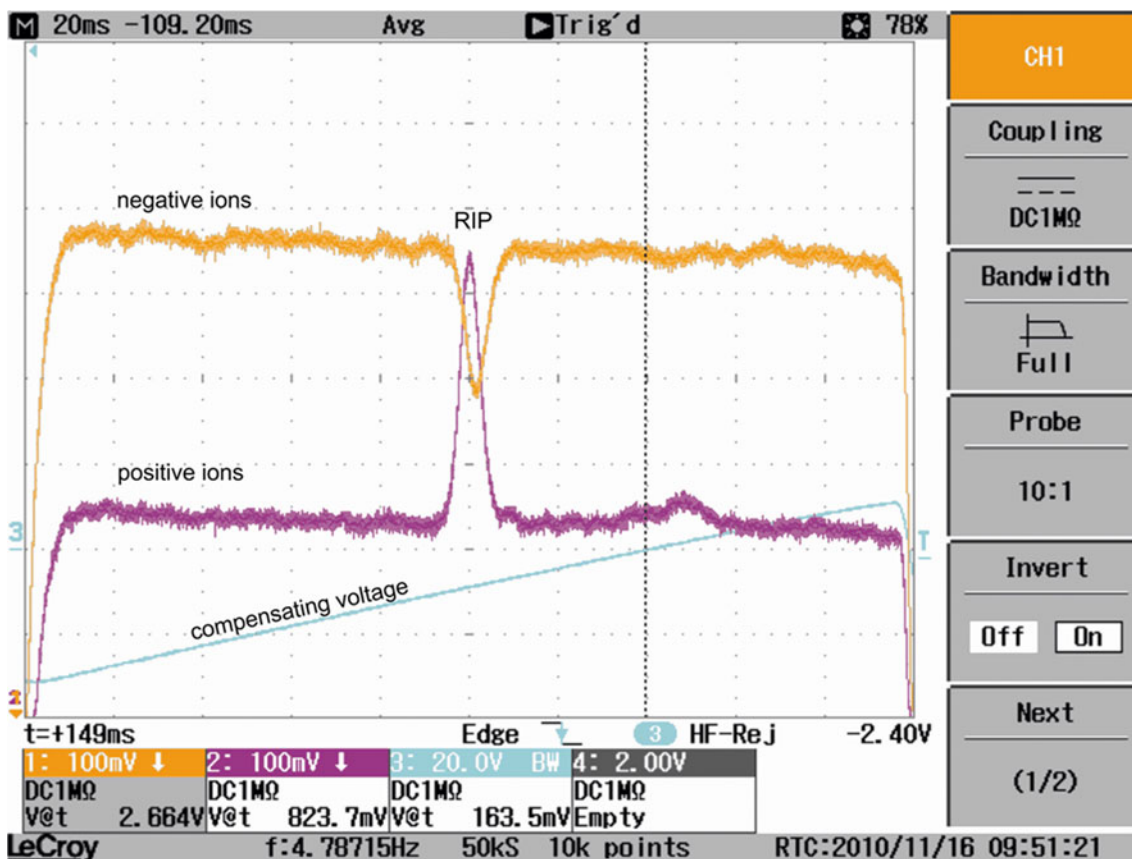


Fig. 9 Reference spectrum air with ammonia dopant



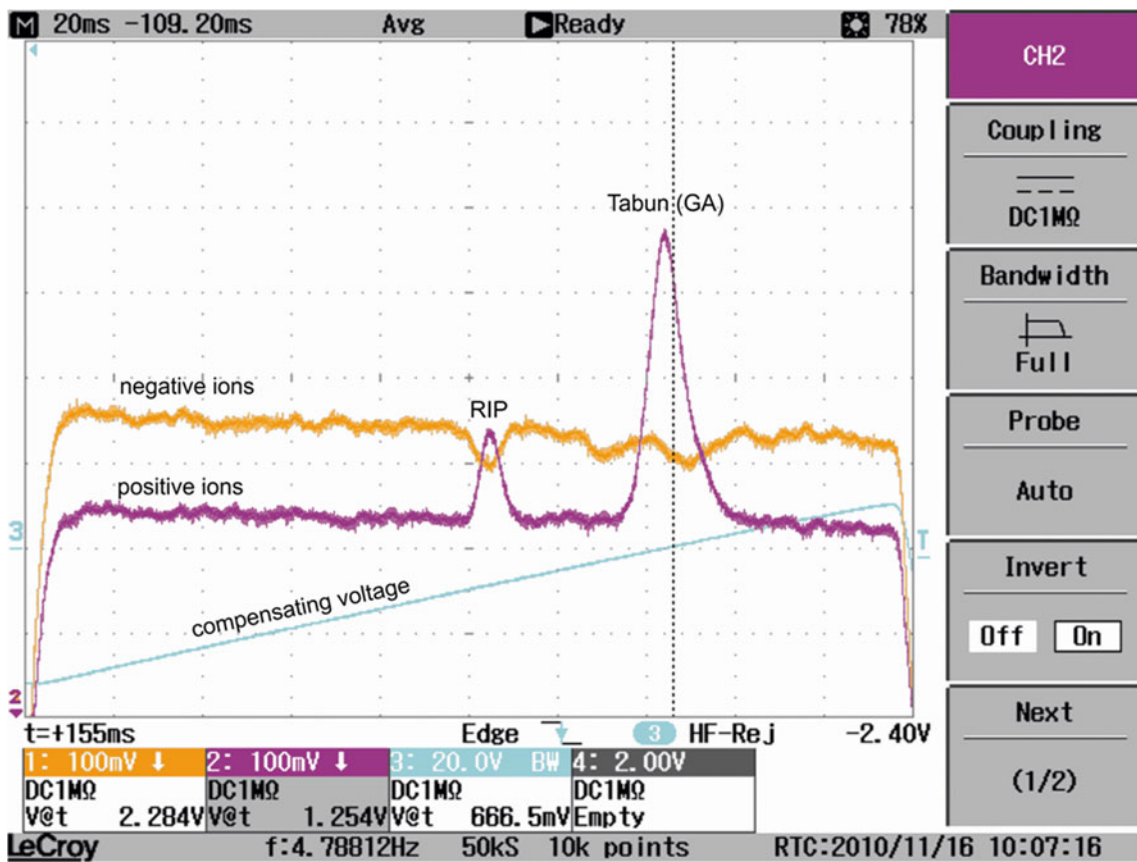


Fig. 10 GA (tabun) detection spectrum

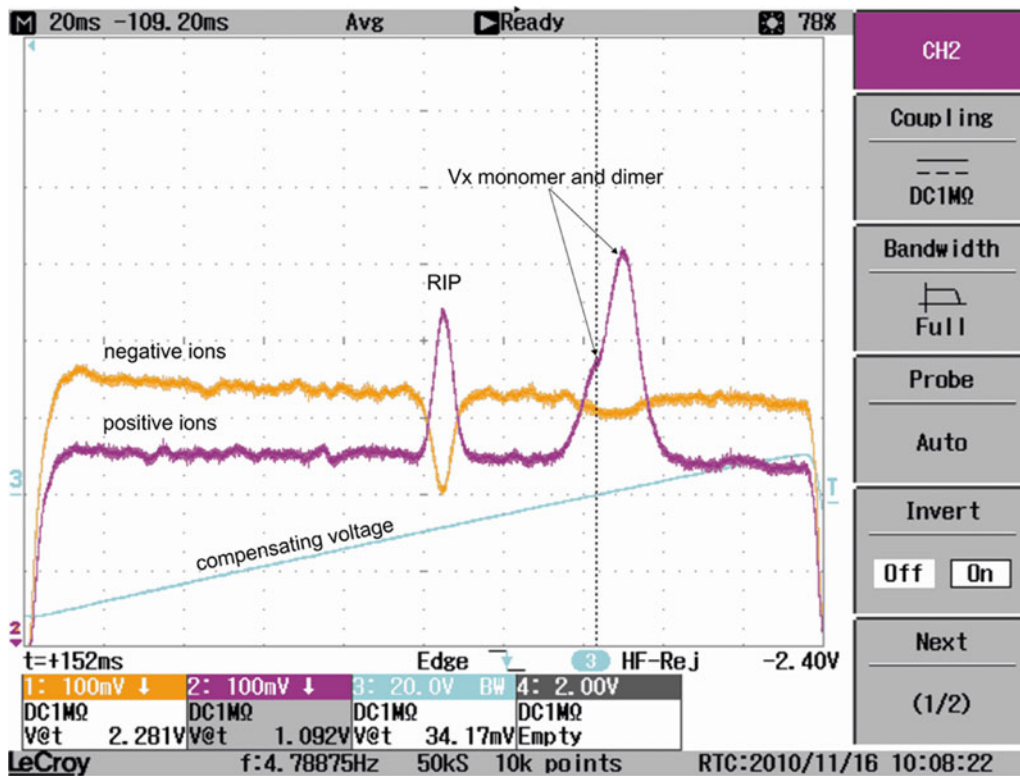


Fig. 11 Vx detection spectrum

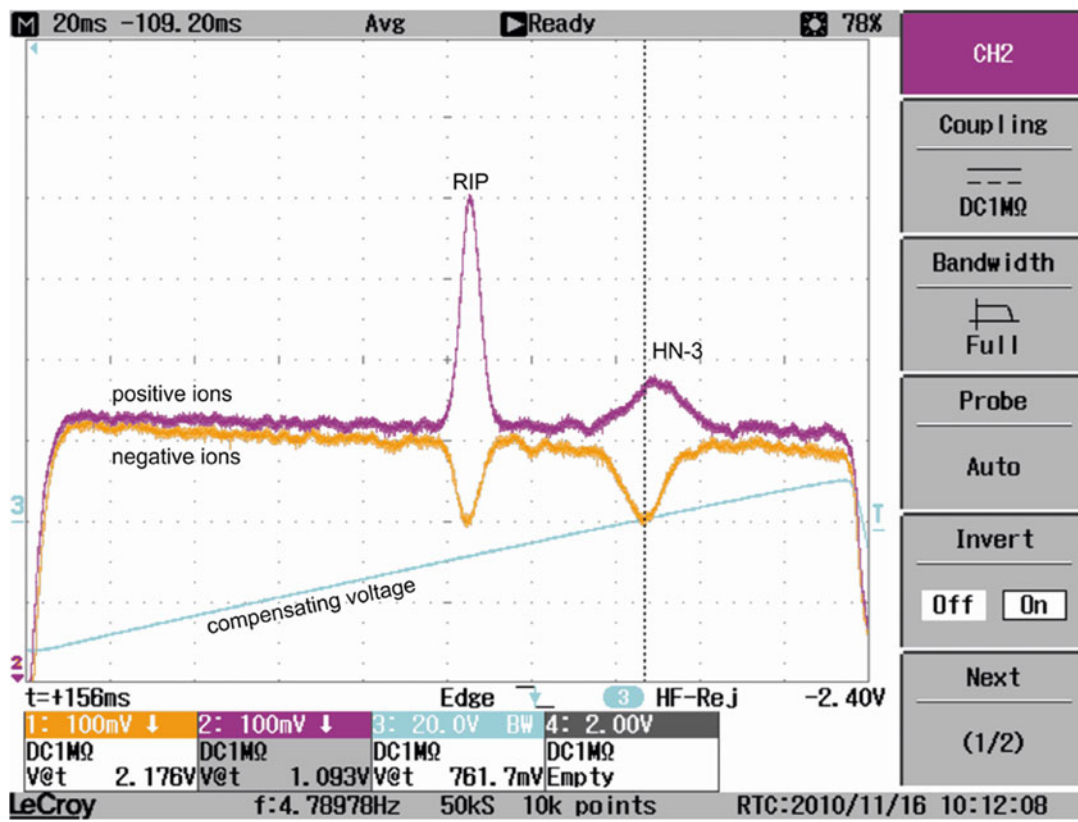


Fig. 12 Mechlorethamine (HN-3) detection spectrum

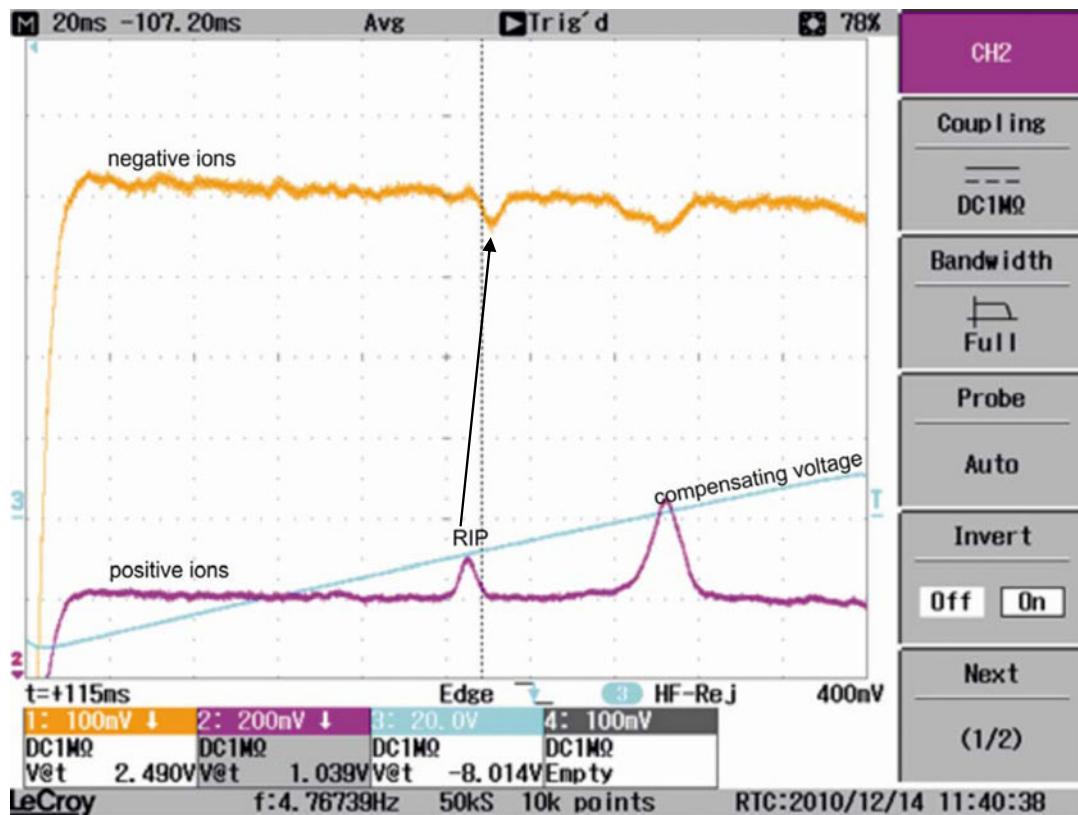


Fig. 13 Spectrum for air

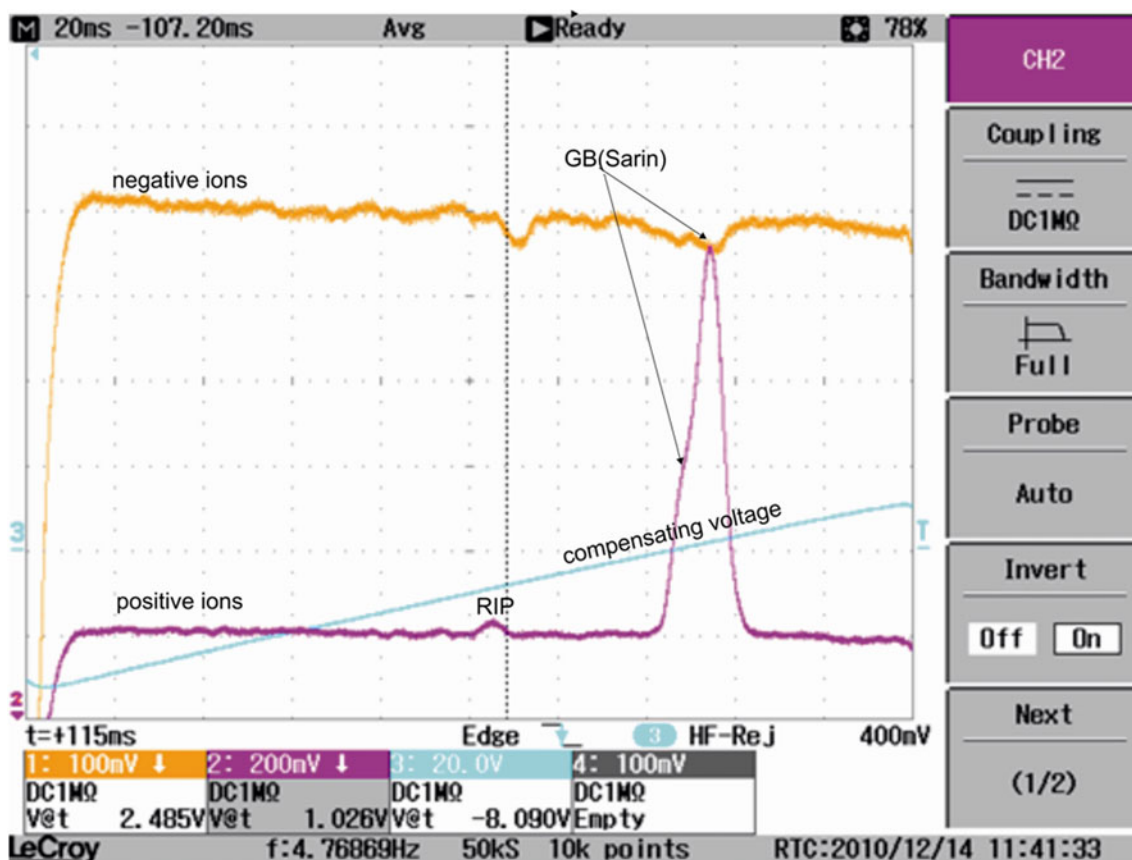


Fig. 14 GB (sarin) detection spectrum

900 mV (it is equal 90 pA ion current). Monomer and dimer are recognizable but practical first is overlapping second one. If we need to detect CWA on very low concentration level following result from above experiment (sarin) point to open gas system as perspective in next applications.

For further analyses “closed” gas set with gas exchanger was chosen. For this case change of mobility,  $\alpha$  – coefficient were calculated (see Table 1).

### HSV Generator (High Speed – High Voltage)

For investigation purposes DMS system described above new high speed high voltage generator was built. See Fig. 15.

On Fig. 16 signal from generator is presented. Ripple of amplitude do not exceed 12 V value. Filling 30 %.

3rd harmonic made pulse deformed and allow fast growth and falling. But 3rd harmonic made unstable work of resonant circuit too. It is shown on Fig. 17 – fluctuation of amplitude voltage.

To determine  $\alpha$  coefficient assumed square wave form signal with 30 % filling at 1 MHz frequency of HSV generator. Amplitude fluctuation of HSV generator caused

Table 1  $\alpha$  - coefficient values and offset voltage for CWA

Analyzed CWA	$\alpha$ - coefficient	Offset voltage – ion type
Sarin GB	-0,0002	+ 0,02 V positive ions
Soman GD	-0,004	+ 0,7 V positive ions
Tabun GA	-0,009	+ 0,1 V positive ions
Vx	-0,013	+ 1,5 V positive ions
Mustard gas HD	+0,013	+0,91 V negative ions
Mechlorethamine HN	-0,00009	+ 0,01 V negative ions
Lewisite L	-0,0066	+ 0,76 negative ions



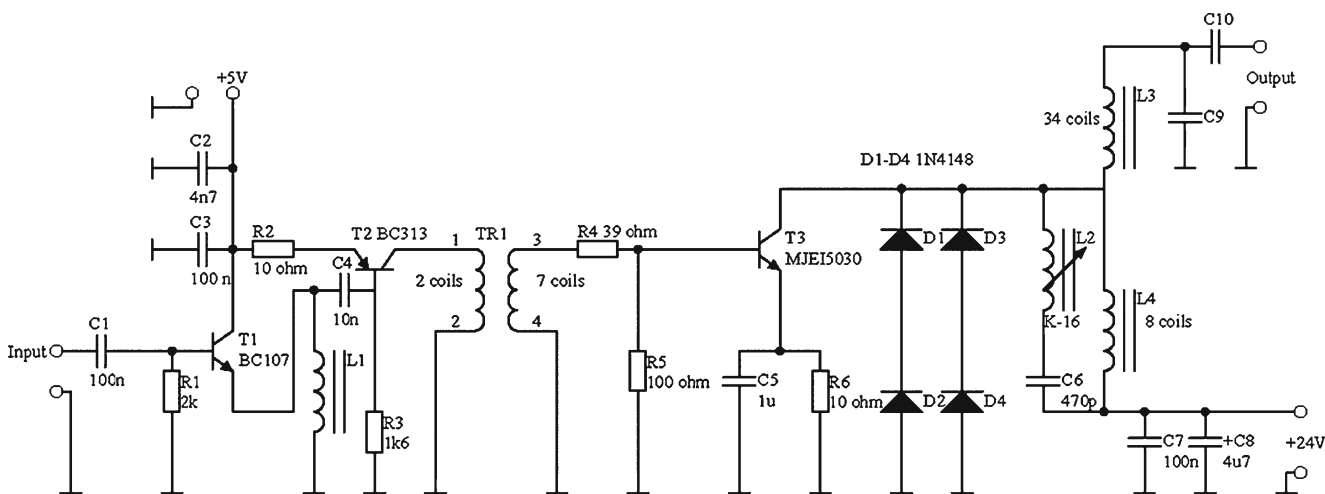


Fig. 15 HSV generator electric wiring diagram

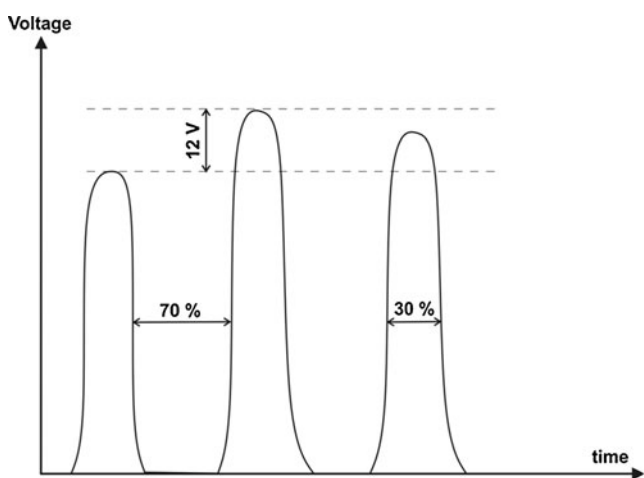


Fig. 16 Signal form HSV generator

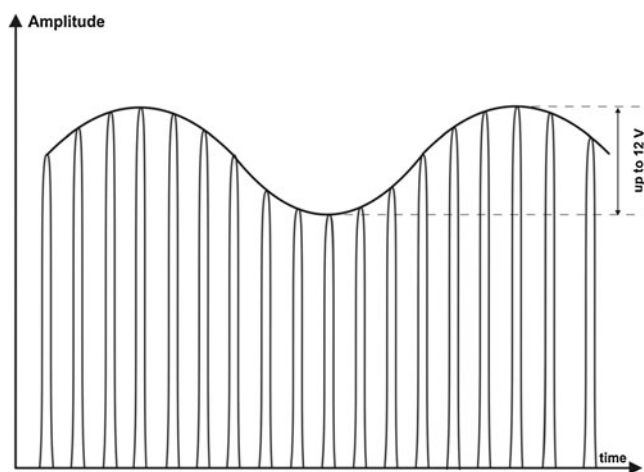


Fig. 17 Amplitude fluctuation of HSV generator

increasing peak width (it makes impossible differentiation between monomer and dimer). However for DMS chamber with 0.25 mm gap and HSV generator with  $f=1$  MHz we observe significant amplitude reduction for ions with reduced mobility above 2.

### Conclusions

During this investigation the authors showed that DMS spectrometer made in thick-film hybrid technology works properly (Fig. 18). It is suitable to detect CW agents on very low concentration (about  $10 \mu\text{g}/\text{m}^3$ ). For  $V_x$  doubled peak was observed like for GB in open gas flow system. Close gas flow system has sensitivity same like linear IMS but open gas flow system is more sensitive and should be subject to next investigation.

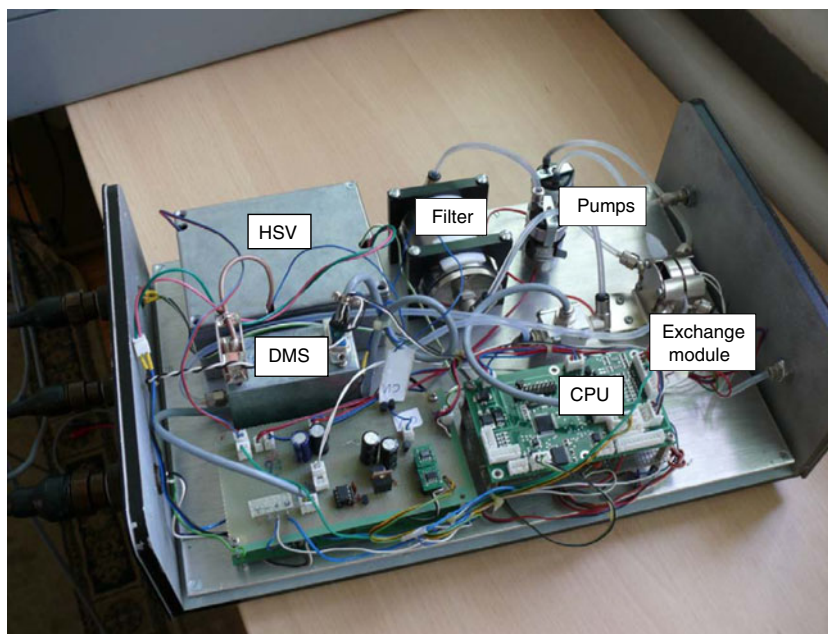
Most CW agents are detected for compensating voltage range from 0 to 1 V. Unfortunately most interference substances is detected in same compensating voltage range.

Concluding - to achieve our target is necessary to:

- Apply open gas flow system,
- Use tuned HSV generator from 250 to 650 V at 2 MHz with better stability (maximum 3 Vpp ripple),
- Calculate full characteristic for monomer and dimer,
- Definite algorithm for separation gas substances.

After this it will be possible to achieve high sensitivity level with low false alarms. For described above DMS model only half of this target was realized. Next pursuit should allow to achieve second scope.

**Fig. 18** Functional model of DMS type spectrometer



**Open Access** This article is distributed under the terms of the Creative Commons Attribution License which permits any use, distribution, and reproduction in any medium, provided the original author(s) and the source are credited.

## References

1. An X, Eiceman GA, Stone JA (2010) A determination of the effective temperatures for the dissociation of the proton bound dimer of dimethyl methylphosphonate in a planar differential mobility spectrometer. *Int J Ion Mobil Spec* 13:25–36. doi:10.1007/s12127-010-0037-6
2. Alexandre A (2009) Shvartsburg differential ion mobility spectrometry nonlinear ion transport and fundamentals of FAIMS. CRC Press Taylor & Francis Group
3. Nazarov EG, Miller RA, Eiceman GA, Krylov E, Tadjikov B. Effect of the electric field strength, drift gas flow rate, and temperature on RF IMS response
4. Eiceman GA, Karpas Z (2005) Ion mobility spectrometry, 2nd edition. Taylor & Francis Group
5. Miller RA, Eiceman GA, Nazarov EG, King AT (2000) A novel micromachined high-field asymmetric waveform-ion mobility spectrometer. *Sensors and Actuators B* 67:300–306
6. Miller RA, Nazarov EG, Eiceman GA, Thomas King A (2001) A MEMS radio-frequency ion mobility spectrometer for chemical vapor detection. *Sensors and Actuators A* 91:307–318
7. Spangler GE, Miller RA (2002) Application of mobility theory to the interpretation of data generated by linear and RF excited ion mobility spectrometers. *International Journal of Mass Spectrometry* 214:95–104
8. Viehland LA, Guevremont R, Purves RW, Barnett DA (2000) Comparison of high-field ion mobility obtained from drift tubes and a FAIMS apparatus. *International Journal of Mass Spectrometry* 197:123–130
9. Reid Asbury G, Wu Ching, Siems WF, Hill HH Jr (2000) Separation and identification of some chemical warfare degradation products using electrospray high resolution ion mobility spectrometry with mass selected detection. *Analytica Chimica Acta* 404:273–283
10. Vinopal RT, Jadamec JR, deFur P, Demars AL, Jakubielski S, Green C, Anderson CP, Dugas JE, DeBono RF (2002) Fingerprinting bacterial strains using ion mobility spectrometry. *Analytica Chimica Acta* 457:83–95
11. Guevremont R, Purves RW, Barnett DA, Ding L (1999) Ion trapping at atmospheric pressure (760 Torr) and room temperature with a high-field asymmetric waveform ion mobility spectrometer. *International Journal of Mass Spectrometry* 193:45–56
12. Eiceman GA (2002) Ion-mobility spectrometry as a fast monitor of chemical composition. *Trends in Analytical Chemistry* 21:259–275
13. Kanua AB, Hill HH Jr (2008) Ion mobility spectrometry detection for gas chromatography. *Journal of Chromatography A* 1177:12–27
14. Cagan A, Schmidt H, Rodriguez JE, Eiceman GA (2010) Fast gas chromatography-differential mobility spectrometry of explosives from TATP to Tetryl without gas atmosphere modifiers. *International Journal of Ion Mobility Spectrometry* 13:157–165
15. Utraiainen M, Kärpänoja E, Paakkanen H (2003) Combining miniaturized ion mobility spectrometer and metal oxide gas sensor for the fast detection of toxic chemical vapors. *Sensors and Actuators B* 93:17–24