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Hexamethyldisilazane (HMDS) detection by Advanced Ion Mobility Spectrometer - AIMS

The ion mobility spectrometry technique offers advantages like high sensitivity (ppb range), fast response (ms range), compact design, operation in atmospheric pressure and ability to separate the isomeric compounds. In this short report we demonstrate the sensitivity and fast response of IMS technique at low ppb level. As a case compound was chosen hexamethyldisilazane HMDS.

The HMDS $C_6H_{19}NSi_2$ of molar mass 161.4 g/mol, is frequently used in various fields like semiconductor industry, photolithography, electron microscopy and many others. The HMDS is toxic irritates skin and eyes and after decomposition can emit highly toxic nitrogen oxide fume. Due to this reasons is monitoring of HMDS in low concentration required especial in clean industrial hall like semiconductor hall are.

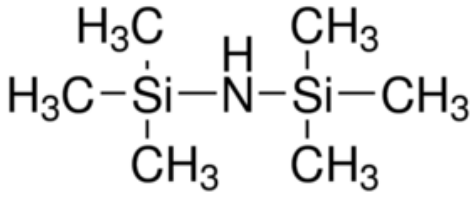

Fig.1. Hexamethyldisilazane

In this short Laboratory Report we demonstrate the ability of Ion Mobility Spectrometer operated in sub-atmospheric pressure for continuous monitoring of HMDS at low ppb level.

Experiment

The **Portable-Advanced Ion Mobility Spectrometer** (**PAIMS**) was used in this experiment. The operating parameters of **PAIMS** are listed in Table 1.

Working pressure	**600 mbar**
Working temperature	**120 ºC**
Drift Gas	**Zero Air**
Drift gas flow	**700 mL/min**
Drift field intensity	**570 V/cm**
Sample gas flow	**60 mL/min**
Polarity	**Positive**

*Table1. **PAIMS** working parameters*

The HMDS of analytical grade purity (Merck) was used in this experiment. The 1mL syringe of HMDS vapors diluted in ratio 1:20 with atmospheric air was used. The syringe was placed to syringe pump (Cronus) and interfaced via 2m long capillary of 0.15mm i.d. to **PAIMS** sample inlet. The long capillary with small i.d. was used in order to prevent diffusion. For calculation of concentration was used vapor pressure 13.8mmHg (PubChem). The **PAIMS** operate in sub-atmospheric pressure and continuous sample sniffing was set to 60 mL/min. The sample inlet suck the atmospheric air, the vapors from the syringe was diluted to sample inlet flow by syringe pump.

Results and discussion

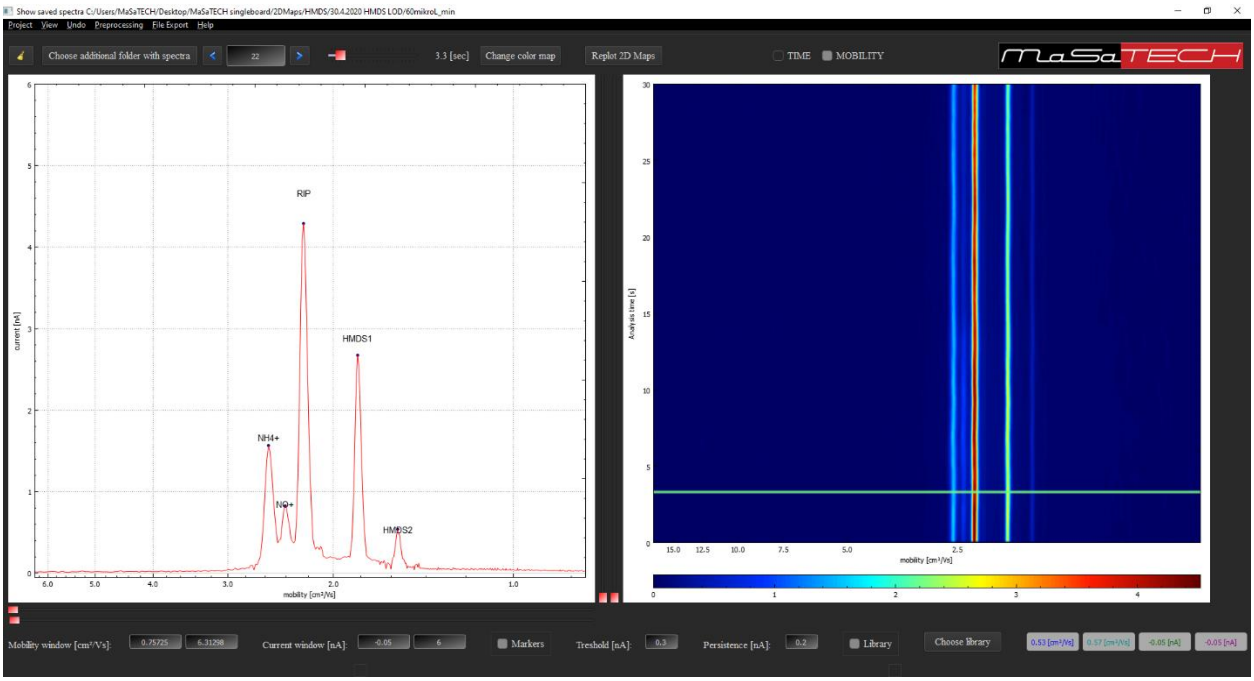

Figure 2. IMS response on 300 ppb of HMDS. Left IMS spectrum measured in reduced mobility mode, Right 2D map of 30s record time

The IMS response on 300 ppb of HMDS is show on figure 2. As we can see from this figure there occur to formation of two peaks: *HMDS1* with reduced mobility 1.82 $cm^2.V^{-1}s^{-1}$ and *HMDS2* with reduced mobility 1.56 $cm^2.V^{-1}s^{-1}$. We suppose that there is going about formation of **protonated monomer** ***H^+.HMDS*** (reduced mobility 1.82 $cm^2.V^{-1}s^{-1}$) and **proton bound dimer** ***$H^+.(HMDS)_2$*** (reduced mobility 1.56 $cm^2.V^{-1}s^{-1}$). The 2D maps on the right side of figure 2 show perfect stability of IMS response during the scan time 30s. During the experiment also longer scan time was tested (20min) and only small deviation bellow 1% in peak intensity was observed.

The **Limit Of Detection** (LOD) for Hexamethyldisilazane was measured directly and was determined 7.5 ppb. Figure 3 shows the IMS response for syringe rate 0.5 µL/min what represents 7.5 ppb.

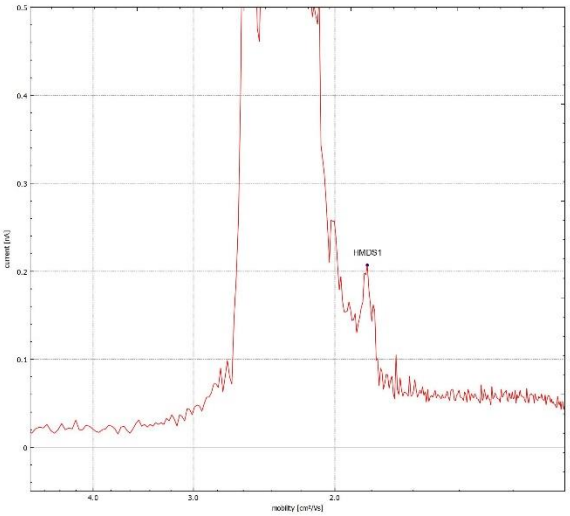

Figure 3. IMS response for 7.5 ppb of HMDS

The MaSa**TECH** software allow calculation of peak **volume**, peak **area,** averaged peak **area** along the monitoring time as well like peak **intensity** and averaged peak **intensity** along the monitoring time. The **PAIMS** results in good dynamic range from 7.5ppb to 1.5ppm as we can see from *Figure 4.*

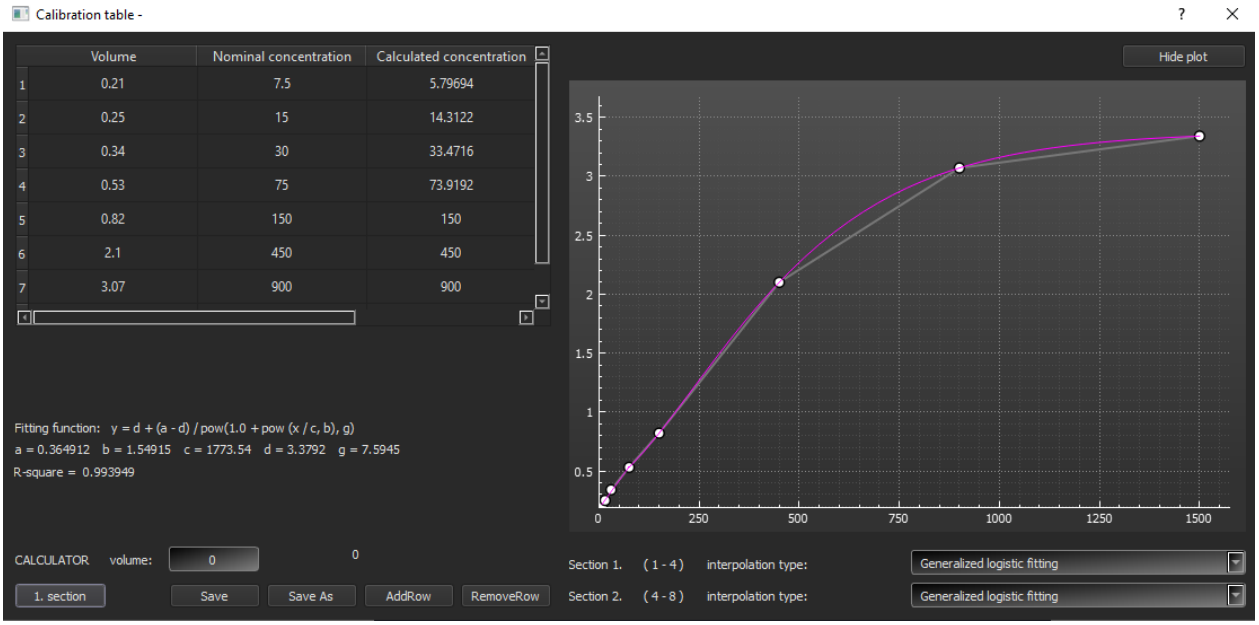

Figure 4. IMS response of HMDS from 7.5ppb to 1.5

MaSa**TECH** software allow us to use several **fitting functions** like linear, exponential, logarithmic, logistic and generalized logistic. For HMDS the *generalized logistic fitting* function seems to be appropriate with R^2=0.9939 .

Fast Response

The main advantage of linear Ion Mobility Spectrometers is related with fast response. The automatic peak derivation and unique measurements in reduced mobility mode allows our instruments fast peak detection and recognition. The online peak derivation giving also **real-time information** about intensity of target peak. The peak intensity can be used for immediate calculation of concentration for each chemical. For more about IMS response on HMDS please see our video:
https://www.youtube.com/watch?v=fUdN7EZIVCo&t=8s