

IMPROVING THE SENSITIVITY OF A MINIATURE MASS SPECTROMETER WITH NOVEL PRECONCENTRATION

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ABSTRACT



A novel preconcentrator is used to increase the sensitivity of a miniature mass spectrometer by several orders of magnitude. The design and method of operating the device allows the concentration of a challenge chemical to be increased without significantly increasing the analysis time of the instrument. The preconcentrator also allows chromatographic separation of a mixture without the need for a GC. An experiment is described in which a miniature mass spectrometer is used to detect low concentrations of xylene with a cycle time of less than 30 seconds.

INTRODUCTION

There is an increasing need for small, affordable, lightweight chemical analyzers. For example, as chemical agent detectors in the field, explosives detectors for transportation, and as fast screening tools in the laboratory. Traditional detectors (e.g., mobility systems) are becoming increasingly unable to provide the specificity, sensitivity, and informing power required for deployment across a wide range of applications.

To overcome the problems associated with traditional lightweight chemical analyzers, 1st Detect has developed a miniature mass spectrometer (MMS-1000™). The MMS-1000 is a desktop cylindrical ion trap mass spectrometer that provides a wide detection range (35 – 500 amu), high resolution (< 0.5 amu FWHM), and fast response time (< 1 second cycle time). The ion trap architecture was chosen because a) there is minimal loss in performance with miniaturization, and b) the ability to perform MS/MS (MSⁿ) analyses without the need for additional hardware. However, miniature mass spectrometers are typically limited to low parts-per-billion sensitivity.

To avoid the problems of integrating a GC column (e.g., high power requirements and slow analysis time), 1st Detect has developed a novel preconcentrator that increases the sensitivity of the MMS-1000 by > 10³ without adversely affecting analysis time. The novel preconcentrator, shown at right, contains a NiCr mesh that is coated with a sorbent material. The choice of material can be customized for broadband detection or specific to targeted compounds. An integrated manifold system, and advanced software control, allows a user to operate the preconcentrator automatically. To operate the preconcentrator, a sample is passed over the coated mesh (typically for 5 – 10 seconds) and analyte is absorbed by the sorbent.

The preconcentrator is then evacuated to the same pressure as the MMS-1000 vacuum chamber and then opened to the vacuum chamber via the manifold (typical evacuation time is 10 seconds). The sorbent is then heated by passing a current through the mesh which desorbs the analyte for analysis by the mass spectrometer. Because the mesh is directly coated with the sorbent, it heats quickly (less than 1 second) which quickly desorbs the analyte into the mass spectrometer thus increasing the apparent concentration by integrating the absorption time. Also, as the analyte is desorbed into a reduced pressure, the concentration gain is also increased by the ratio of the absorption and desorption pressures.



EXPERIMENTAL

To verify the performance of the preconcentrator, the MMS-1000 was configured to operate with a cycle time of 1 second (which is 10 x 100 msec 'microscans') and the mass range set at 360 amu. A sample was prepared by injecting 0.025 ml of xylene into a 50 litre Tedlar bag and allowing the analyte to evaporate, resulting in a concentration of 91 ppb. A sample (1 litre) of the mixture was then introduced to a second 50 litre Tedlar bag containing 49 litres of dry nitrogen, resulting in a concentration of 2 ppb.

The 2 ppb sample was first introduced to the MMS-1000 across the membrane inlet (flow rate = 600 ml/min) and the resulting spectrum recorded. The sample bag was then moved to the preconcentrator inlet, the preconcentrator operated as the sample flowed over the preconcentrator (absorption time was set to 10 seconds). The spectrum was recorded as the preconcentrator was desorbing the analyte.

RESULTS

Figure 1 (left) below shows the resulting spectrum as the sample was introduced across the membrane inlet. It can be observed that there is no appreciable ion current. Figure 2 (right) shows the recorded spectrum as the same sample was introduced across the preconcentrator. It can be observed that there is a significant increase in the signal with well formed, high resolution peaks resulting.

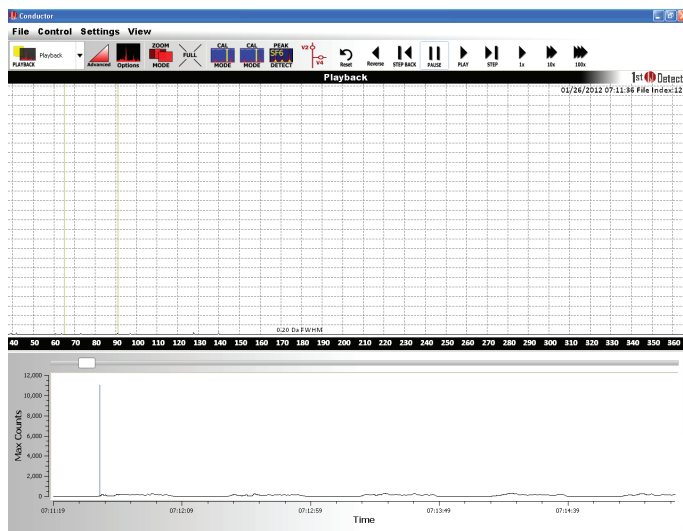


Figure 1 - Spectrum recorded as sample was introduced across membrane inlet

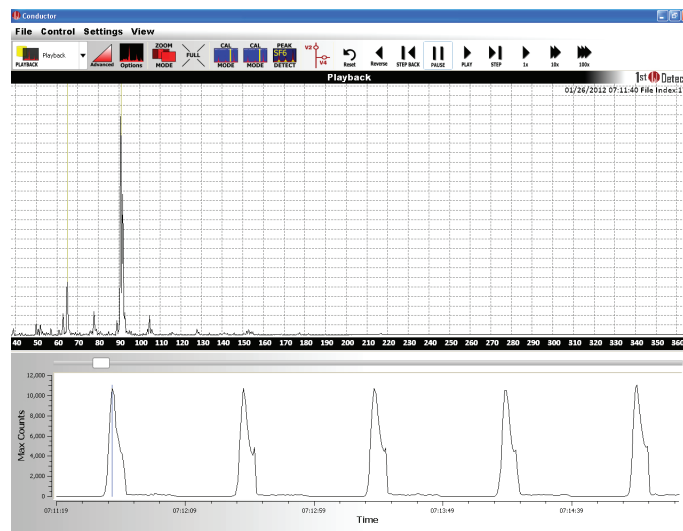


Figure 2 - Spectrum recorded as sample was introduced via preconcentrator

CONCLUSION

The authors demonstrated the increase in sensitivity of a miniature mass spectrometer enabled by a novel preconcentrator. Samples with concentrations as low as 2 ppb were measured with a cycle time of less than 30 seconds. The combination of high performance analysis enabled by the MMS-1000 mass spectrometer, and high sensitivity enabled by the preconcentrator, demonstrates that mass spectrometry can be a viable solution to the needs of desktop and portable chemical detection.

References:

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