

Towards smaller and faster gas chromatography–mass spectrometry systems for field chemical detection

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Abstract

Gas chromatography–mass spectrometry (GC–MS) is already an important laboratory method, but new sampling techniques and column heating approaches will expand and improve its usefulness for detection and identification of unknown chemicals in field settings. In order to demonstrate commercially-available technical advances for both sampling and column heating, we used solid phase microextraction (SPME) sampling of both water and air systems, followed by immediate analysis with a resistively heated analytical column and mass spectrometric detection. High-concern compounds ranging from 140 to 466 amu were analyzed to show the applicability of these techniques to emergency situations impacting public health. A field portable (about 35 kg) GC–MS system was used for analysis of water samples with a resistively heated analytical column externally mounted as a retrofit using the air bath oven of the original instrument design to heat transfer lines. The system used to analyze air samples included a laboratory mass spectrometer with a dedicated resistive column heating arrangement (no legacy air bath column oven). The combined sampling and analysis time was less than 10 min for both air and water sample types. By combining dedicated resistive column heating with smaller mass spectrometry systems designed specifically for use in the field, substantially smaller high performance field-portable instrumentation will be possible.

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1. Introduction

Many fieldable rapid detection and identification methods for chemicals of high concern to military forces rely on color changes to a chemical-impregnated paper for liquid samples, or to so-called “detector tubes” for air samples. Rapid detection in water samples is also available through military test kits based upon chemical reactions that produce visible color changes. These colorimetric methods, while relatively easy

to use in the field, offer only tentative chemical identification and poor sensitivity.

Widely fielded instrumental techniques for detecting this type of chemical include systems based on ion mobility spectrometry and a man-portable system that combines a gas sampler with a gas chromatography–mass spectrometry (GC–MS) instrument. Ion mobility spectrometry-based methods are not compound-specific and offer poor quantification dynamic range. The widely available man-portable GC–MS instrument (with an inseparable sampler) can provide compound-specific data. The combined GC–MS/sampler system provides a sampling device of 16 kg, and offers sampling/analysis times of around 15–20 min with

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chromatography performance (e.g. peak shapes and widths) generally inferior to laboratory instrumentation. Both of these instrument systems are limited to gas phase analytes unless additional hardware is used.

Laboratory-based mass spectrometry is a mature analysis method that has become an indispensable tool for researchers worldwide. Large mass spectral libraries are available when 70 eV electron impact ionization mass spectrometry (EIMS) is used. This allows detection and identification of trace levels of many mixture components when a separation method is used prior to examining the clean spectra produced from the separated compounds. Even sub-optimal separation can allow deconvolution software to assist in identifying unknown chemicals present when EIMS detection is used. For EIMS analysis, the separation method of choice is typically GC. In addition to the usefulness of existing mass spectral libraries, proven laboratory-based MS instrumentation and highly developed and robust open tubular GC columns are available at reasonable cost.

Besides the use of GC, other inlet methods are available to support the use of mass spectrometry in field settings. These inlet methods range from simple to complex: with a pinhole inlet, atmosphere gases give a high background and shorten the mass spectrometer filament life when EIMS is used; membrane inlet methods provide some analyte discrimination and help reduce the presence of atmosphere gases [1], but are problematic for detection and identification of trace level components in complex mixtures. An ion trap MS system can perform MS/MS analyses, effectively retaining target analyte ions in the trap with confirmatory daughter spectra provided [2]. However, MS/MS used without a prior separation step is typically selective for expected analytes, and ions resulting from unanticipated compounds would be retained in the trap only by chance.

The ability to separate complex mixture components in time prior to mass spectrometric detection offers the unambiguous ability to complete spectrum matching of clean EIMS spectra. Eckenrode [3] describes environmental and forensic applications of field-portable GC–MS and makes the point that field GC–MS is useful for situations where rapid analyte identification, and where a high degree of certainty are required. In 1994, McDonald et al. [4] reviewed and discussed the state-of-the-art GC–MS instrumentation then available and useful for completing analysis in field settings. Ten years later, essentially all field-portable GC–MS instruments commercially available and capable of analyzing the full range of compounds traditionally expected from a GC–MS instrument still rely on the traditional column heating method used in most laboratory instruments: air bath heating.

The costs associated with adding a GC separation step to mass spectrometry in field settings include the added weight, complexity, and power consumption of the resulting sampling and detection hardware. In order to make gas chromatography more compatible with EIMS in field settings, sampling and column heating methods are needed that significantly improve upon those typically used. This should allow rapid

detection of extremely hazardous chemicals from the standard environmental matrices (air, water, and soil), with relatively little sample handling and preparation. An ideal fieldable GC–MS chemical detection/identification system must have the capability to rapidly sample, detect, and identify a wide range of high concern chemicals from a variety of sample matrices. The footprint of such a system must be small, power consumption and weight must be low, and it should be easy to use.

In this work, we used solid phase microextraction (SPME) to sample five dangerous chemical compounds and a high molecular weight fungal toxin as water contaminants, and four dangerous chemical compounds with appreciable volatility as air contaminants. The presence of any of these chemicals in water supplies or as air contaminants would be of public health concern. For both air and water samples, a low thermal mass (LTM) GC column with resistive heating was used to separate the sampled compounds prior to mass spectrometric detection. Two instruments were used: for water samples, a field-portable GC–MS system was used. This system weighs about 35 kg, and the LTM column assembly was retrofitted to the exterior of the instrument's isothermally-heated air bath oven. As an example of a GC–MS system where the entire air bath GC oven was omitted and LTM column heating was engineered by design, SPME samples from contaminated air were analyzed using a typical commercial production mass spectrometer. For this instrument, rather than retrofitting the LTM GC column assembly to an existing air bath oven, the LTM GC column was interfaced directly to the mass spectrometer through a small heated box that contained the GC injector and the transfer line into the mass spectrometer.

The use of LTM GC column heating technology and a sampling method such as SPME that allows rapid sampling of air, water, and soil matrices point towards improvements in both equipment systems and sampling/analysis methods that will allow reductions in the size and weight of a GC–MS instrument with excellent performance. High sample throughput and the ability to detect and identify compounds with widely different physical properties can be demonstrated with the sampling methods and instrumentation systems described in this work.

2. Materials and methods

2.1. SPME sampling

The SPME fibers and holder used are commercially available from Supelco (Bellefonte, PA). The fiber coating used for water samples was the polyacrylate type (PA, 85 μm coating thickness), as it is capable of withstanding injector temperatures up to 315 °C. A high injector temperature is necessary to desorb the large T2 mycotoxin compound present in water samples [5]. For air samples, the SPME fiber coating was polydimethylsiloxane/divinylbenzene (PDMS/DVB,

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