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EVALUATION OF A PORTABLE GC/MS FOR SCREENING ORGANIC CONTAMINANTS IN SOIL

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ABSTRACT

In an effort to speed up clearance of building-construction sites in the Edgewood Area of Aberdeen Proving Ground, Maryland, Argonne National Laboratory leased a Bruker Instruments Mobile Environmental Monitor (MEM) to analyze for organics in soils. The MEM is a portable gas chromatograph/mass spectrometer (GC/MS). Argonne's MEM was mounted in a Chevy Blazer and driven to each site of interest. The Bruker analytical system uses a "probe" with a thermal devolatilization ring on the end and short GC column inside to permit rapid assessment (via GC/MS) of characteristic fragmentation masses in small samples of soil as soon as they are removed from the ground. Volatiles and semivolatiles can be qualitatively determined with the probe and MS. Quantitation of volatiles and semivolatiles is handled by substituting a GC oven (and its contained 30-m-long GC column) for the probe.

Argonne evaluated the MEM over a nine-month period, during which

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176 soil samples were analyzed by probe and 124 by oven. Argonne's experience with the Bruker MEM indicates that some features of the system are at present in the developmental stage. The software is cumbersome and does not yield results suitable for convenient reporting. Instrument calibration for determination of volatiles and semivolatiles cannot be done consistently and is therefore time consuming. Downtime due to loss of instrument sensitivity or malfunction of parts was judged excessive. The MEM is best used to rapidly screen soil samples (by probe) for a large number of volatile and semivolatile compounds. The qualitative results can then be used to determine more comprehensive and detailed sampling and analysis procedures.

INTRODUCTION

Argonne National Laboratory is currently working with the Baltimore District of the U.S. Army, Corps of Engineers, to develop the most efficient and cost-effective techniques for screening large numbers of soil samples for a wide variety of volatile and semivolatile organic compounds. A Bruker Instruments "MEM" (mobile environmental monitor) was evaluated for nine months to see how useful it would be for rapid characterization of soil samples. The desirability of field GC/MS, and its accomplishment using the Bruker MEM system, has been described by Kowalski (1991) and Hadka and Dickinson (1988).

The MEM is a mass spectrometer with two sample inlet mechanisms; (a) a 3.5-m "probe" equipped with a heated sample head for thermal volatilization of organics in soil samples, and with a short capillary column

for a small amount of chromatographic separation and (b) a gas chromatograph oven with a 30-m capillary column for full chromatographic separation of compounds. Both inlet mechanisms were employed during Argonne's investigations a the Edgewood site.

Argonne's experience with the Bruker MEM indicates that the instrument is a useful tool for screening soil samples for a wide variety of organic chemical contaminants. The power of the mass spectrometer and the unique user interface makes real-time soil characterization possible. Argonne found, however, that several factors presently limit the MEM's capacity to function as a mobile GC/MS for quantitation of organic compounds.

SYSTEM DESCRIPTION

Argonne's Bruker MEM instrument is equipped with built-in battery power, is resistant to shocks, and will operate from -3°C to 50°C with no external cooling or heating requirements. The mass spectrometer has a mass range of 1-400 Daltons, which minimizes power consumption. It can operate for 8-10 hours on battery power, or indefinitely using a generator or conventional AC power. An MS-DOS 386-based data system can be used to acquire, analyze, and archive all GC/MS data. Sampling accessories are available for a wide range of monitoring situations: the MEM probe with a 3.5-m GC capillary column is used for thermal desorption of organics from a soil matrix. A temperature-programmable GC oven with a capillary column is also available. Both inlet mechanisms operate with ambient air

as the carrier gas, thus eliminating the need for external carrier-gas cylinders.

OPERATION

Analysis of soil samples at a potential construction site is done directly using the MEM probe with no sample preparation. The instrument's mobility and the 3.5-m reach of the MEM probe make it possible to analyze most soil samples within a few minutes after recovery. In this method, several grams of soil are directly analyzed by pressing the hot MEM probe tip against a sample. The 250°C temperature of the probe tip rapidly volatilizes most organic compounds, which are then sucked into the 3.5-m capillary column of the MEM probe. Organic compounds are detected by the mass spectrometer and displayed on the MEM monitor. The monitor can display the data generated by the mass spectrometer in several formats. In the air monitor format, a bar graph charts the intensity of up to four characteristic fragmentation masses of each compound of interest (up to 22 compounds can be continuously monitored). This format was used to screen soil samples at the sampling sites.

The data generated in the air-monitor format are qualitative and can often indicate contamination while not specifically identifying particular compounds. Therefore, it is often necessary to substantiate or elaborate on the air-monitor data with more detailed analysis by GC/MS. A small GC oven can be attached to the MEM (in place of the probe) to obtain quantitative results. The GC oven and its 30-m-long column enables the

MEM to more completely separate the organic compounds and thereby more accurately quantify concentrations and identify unknowns. Two standard sample preparation techniques are employed in conjunction with the GC oven:

1. Volatile compounds are extracted from a soil sample with a purge-and-trap technique. The trap consists of a glass tube filled with Tenax and silica gel adsorbents. The compounds are thermally desorbed off the adsorbent and introduced into the GC oven in the desorption port.

2. Semivolatile compounds are extracted from a small soil sample with a methanol solvent-extraction procedure. The soil is mixed with a small volume of methanol and shaken. An aliquot of the extract is then injected onto a glass injection liner and thermally desorbed into the GC oven in the desorption port.

APPLICATIONS

Several MEM features are unique and technically advanced. First when the instrument is configured with batteries, it fits easily into the back of a vehicle (i.e., Blazer or other similar vehicle) and is therefore portable. Second, the MEM does not require a special carrier gas (air is the carrier gas) and therefore there is no need for gas cylinders. Third, the instrument has rugged construction, making it field durable. Fourth, the

heated probe head allows for rapid identification of semivolatile as well as volatile organic compounds.

Argonne personnel made a total of 300 analyses with the MEM over a nine-month period (see Table 1) and found that the MEM was best suited for field screening of soil samples using the probe as the inlet mechanism. The bulk of the use of the MEM was within the Edgewood Area of Aberdeen Proving Ground, MD; a small effort was conducted in Johnson City, NY, to screen for contaminated soils at Air Force Plant 59. The relative abundance of contaminants in soil samples can thus be determined rapidly in the field. Samples can be selected for more detailed analysis based on the results of the probe screening. In some instances, contaminants can be indentified and semiquantified in the field if they occur in a relatively "clean" matrix.

Table 1. MEM Soil Analyses at
Edgewood Area, Aberdeen Proving Ground, Maryland,
and Air Force Plant 59, Johnson City, New York

Project No.	Probe Analysis	VOA Oven Analysis	SVOA Oven Analysis
E162	25	7	18
U531	56	4	14
CR02	25	4	7
A003	4	1	3
A087	15	5	9
F572	17	3	5
A604 (3726)	16	10	67
SUB TOTAL	157	34	67
AFP-59	19	4	19
TOTAL	176	38	86

LIMITATIONS AND RECOMMENDATIONS

As with most portable analytical equipment, portability and size reduction is often achieved at the expense of versatility. Argonne found that the quantitation techniques used with the GC oven were somewhat cumbersome and time consuming and, therefore, believed best suited for a laboratory environment.

The MEM system can be improved in two main areas. First, a more precise method of sample introduction should be available for the GC oven. Difficulty in obtaining consistent calibration runs may be due, in part, to the current glass liner desorption procedure. Second, an improved data-processing software package is needed. The software should maintain

project data in a more useful form. The software system should allow the operator to perform more conveniently machine-turning and monitoring functions, calibration runs, and report-generation functions in an integrated, user-friendly manner.

SUMMARY

The unique features of the MEM make it a useful instrument for field screening of soil samples for organic chemical pollutants. It is capable of rapidly screening soil samples for a large number of volatile and semivolatile compounds. The results are qualitative and are best used to scope out more comprehensive and detailed sampling and analysis strategies. Argonne found that the quantitation of chemical compounds is more reliably and productively determined using laboratory GC/MS equipment.

DISCLAIMER

endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

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