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## On-site environmental analysis by membrane extraction with a sorbent interface combined with a portable gas chromatograph system

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A novel device, membrane extraction with a sorbent interface (MESI) coupled with a portable gas chromatograph (GC) system, has been developed. The main components of this system include a membrane module, a microtrap, and a control unit for the heater and cooler. The membrane module, as an on-line sample-introduction device for this system, can be manipulated in different configurations, allowing for the selective permeation of analytes across the membrane into the carrier/stripping gas. The analytes are trapped and concentrated onto a microtrap, which serves as an injector for gas chromatography separation. A concentration pulse of the trapped analytes is generated through direct electrical heating of the microtrap. The characteristics of this system have been explored, and its applicability and effectiveness have been demonstrated in field monitoring applications including the analysis of toluene in wastewater, Volatile organic compounds (VOCs) in laboratory air, and chloroform in swimming-pool water. This system is very promising, as it is a simple, fast, and portable tool for on-site process environmental monitoring.

**Keywords:** Membrane extraction; Sorbent interface; Portable gas chromatography; Continuous monitoring; Volatile organic compounds

### 1. Introduction

On-site chemical analysis is becoming increasingly important due to both the growing knowledge of the toxicity of various chemicals and the continuous increase in the regulations of environmental legislation driven by increasing public awareness of environmental problems. Hence, there is a critical need for instrumentation that can be used to carry out automated and on-line/on-site fast analysis to provide accurate information on a continuous basis. This information can then be used for process control, while meeting regulatory compliance. Currently, there are a variety of analytical techniques, such as Fourier-transform infrared spectrometry, X-ray fluorescence spectrometry, and mass spectrometry, which are used for continuous and on-line monitoring [1–3].

In general, spectroscopic techniques are ideal for process monitoring because of their rapid analysis speed. However, they also have many disadvantages. For instance, water

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vapour, which commonly exists in samples, can seriously interfere with analysis results. In addition, individual organic compounds in complex mixtures are difficult to identify, owing to the overlapping of absorbance bands from the different compounds [4]. Mass spectrometers (MSs) face similar challenges, such as deconvolution of individual spectra in a complex matrix and interference from water and carbon dioxide from the atmosphere, if monitoring in high-humidity sites. Moreover, both spectroscopic and MS techniques are quite expensive.

Gas chromatography is an excellent technique for on-site environmental monitoring. It has been used in continuous monitoring applications, including process monitoring and the screening of food, environmental, and biological samples since the 1950s [5]. Portable gas chromatographs (GCs) were invented as a simpler and less expensive alternative to laboratory models. Most often, short capillary columns operated at high carrier gas velocities are used in this kind of instrument, making a portable GC capable of performing high-speed separations. The attached gas generator in a portable GC avoids the inconvenience of transporting additional gas tanks into the field, eliminates the inconvenience of transporting the samples to the laboratory, and minimizes the formation of sample artefacts. Therefore, a portable GC is an ideal tool for on-site analysis.

Although GCs with normal detectors, such as the flame ionization detector (FID) or the thermal conductivity detector (TCD), possess a high sensitivity, conventional GCs are incapable of doing reliable measurements of ultra-trace amounts of target compounds [6]. For example, carcinogenic compounds, such as bis (chloromethyl) ether require detection levels at the parts per trillion level, because its maximum acceptable concentration is 0.1 ppb [7]. However, these low detection levels cannot be attained with normal detectors. Some more sensitive detectors, such as the photo-ionization detector (PID) and the electron-capture detector (ECD), were developed as an alternative. However, they usually respond to specific properties of analytes and do not act as a general detector for all target compounds.

Another basic method to improve sensitivity of a GC is to increase the injection volume. However, this method has certain limitations in trace analysis. A large volume injection requires a long injection time, which causes excessive band broadening, especially in capillary columns. Furthermore, a sample valve intermittently injects an aliquot of sample from the process stream, and no information is available in the period between two injections. This can be a serious limitation for monitoring processes that change rapidly with time.

To overcome these shortcomings, various preconcentration methods have been introduced during sample preparation steps prior to GC analysis. Traditional preconcentration methods, such as liquid-liquid extraction (LLE) or solid-phase extraction (SPE) are discrete and laborious, and involve multi-step procedures that lead to the loss of target compounds, cross-contamination, and degradation, and most often, require the use of toxic and expensive solvents. These analytical procedures are usually time-consuming and unsuitable for on-site real-time analysis. Other methods such as 'purge and trap' have been used to preconcentrate the target compound. However, purge and trap requires that the sample be weighed and then transferred to a purge vessel, thus increasing the potential for analyte loss. A very simple and efficient sample-introduction technique that has been successfully used for fast field analysis is called solid-phase microextraction (SPME) [8]. However, this cannot be used for semi-continuous monitoring without additional complicated instrumentation, such as

a modified autosampler [9]. These additional pieces of mechanical equipment are not desired in field analysis.

As an alternative to SPME, membrane extraction with a sorbent interface (MESI) has been developed [10]. The MESI combines sampling, preconcentration, and sample introduction in one procedure, eliminating the traditional steps that cause sample loss and contamination. It is a promising sample-preparation technique for either routine analysis or long-term continuous monitoring of analytes in various sample matrixes. This technique can be used without modifying the portable GC. It is an excellent method for field monitoring, since it provides on-line sample preconcentration, and the portable GC offers mobility and convenience for on-site monitoring. The MESI in combination with portable GC (MESI-portable GC) system is described herein, the characteristics of this system are described, and several applications of MESI-portable GC for continuous analysis of environmental samples are reported.

## 2. Experimental

### 2.1. Materials and procedures

Benzene, toluene, ethylbenzene, xylene, acetone, and chloroform were purchased from Sigma-Aldrich (Mississauga, ON, Canada). Two kinds of commercial microtraps (Tenax and Carboxen) were provided by Restek Corp. (Bellefonte, PA). The microtraps were conditioned under nitrogen at 200°C for 2 h before being used. Polydimethylsiloxane/divinylbenzene (PDMS/DVB) fibre (65 µm) was purchased from Supelco (Bellefonte, PA). A standard gas generator and permeation tubes (Kn-Tek labs, Texas city, TX) were used to generate a standard gas mixture stream of toluene, ethylbenzene, and xylene [11]. Standard chloroform gas was obtained in the dynamic mode by using a 'liquid injection' standard gas generation system [12].

MESI-portable GC continuous on-site monitoring was performed with a portable model 8610C GC (SRI Instruments, Torrance, CA). The analysis parameters are listed in the captions of the figures presented herein. The SPME coupled with gas chromatograph and mass spectrometry (SPME-GC-MS) analyses for chloroform identification were performed using a Varian 3400 GC (Varian Canada Inc., Mississauga, ON, Canada) coupled with an ion-trap detector. This was equipped with an Rtx-5MS capillary column (30 m × 0.25 mm<sup>2</sup>, 0.25 µm film thickness) (Restek Corp., Bellefonte, PA). Helium (TAG grade, Praxair, ON, Canada) was used as the carrier gas at 12 psi. The initial column temperature was 35°C, then held for 3 min, and subsequently increased at a rate of 10°C/min to 250°C and held for 5 min. The MS spectra were recorded with the electron ionization (EI) mode, with a scan range of 50–650 amu at a scan frequency of 1 scan/s. The filament emission current was 10 µA. The transfer line temperature was 260°C. Eluants were identified using a NIST 98 spectra database.

### 2.2. MESI-portable GC system

A general scheme of the MESI-portable GC system used in this study is illustrated in figure 1. The main components of this system include the membrane module, the microtrap (sorbent interface), the control unit of the cooler and heater, the

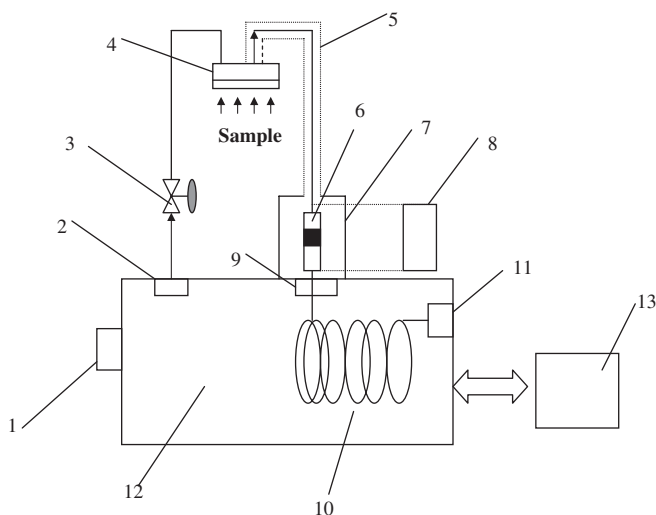


Figure 1. Schematic diagram of the MESI-portable GC system: 1: attached hydrogen generator; 2: pressure-regulation valve; 3: flow controller; 4: membrane module; 5: heating tape for sample transfer (if necessary); 6: microtrap; 7: control unit for heater and cooler; 8: power supply for control unit; 9: GC injector; 10: capillary column; 11: detector; 12: GC oven; 13: computer.

GC, and the system control computer. The design of the membrane module was presented in a previous work [13]. A flat-sheet membrane (50  $\mu\text{m}$ , SSP-M100, dimethylsilicone, Membrane Components, Ballston Spa, NY) was not self-supported and therefore required a special holder to support it. One side of the membrane was exposed to the sample, and the other side was exposed to the carrier/stripping gas. Teflon washers and stainless-steel plates were used on both sides of the membrane. A fine stainless-steel wire mesh, mounted in a stainless-steel plate, supported the exterior of the membrane that was exposed to the sample. Both plates were fastened with 12 screws. The carrier/stripping gas was supplied to the module through Silicosteel<sup>®</sup>-treated tubing (0.160 inch o.d., 0.010 inch i.d., Restek Corp.) that was fitted tightly into the Teflon washer. The carrier gas pressure lifted the membrane and allowed the free passage of the gas to the outlet tube on the opposite side of the membrane surface. After leaving the membrane module, the carrier gas and analyte proceeded to the microtrap. A schematic representation of the microtrap design was shown in a previous work [13]. Microtraps for MESI were made from a small-diameter Silicosteel<sup>®</sup>-coated tubing (5 cm  $\times$  0.75 mm i.d.) packed with traditional trapping material, such as Carboxen, Tenax, or other divinylbenzene resins. The bed length was 1.7 cm, and the sorbent was 60/80 mesh. Quartz wool was placed at the ends of the sorbent bed to retain the packing. At both ends, the microtrap tubing was fitted into the system using two deactivated Silicosteel<sup>®</sup>-treated zero-dead-volume connectors (0.125 inch i.d., Restek Corp.) and four Supeltex/TM graphite ferrules (Supelco, Bellefonte, PA). A connection between one end of the microtrap and the membrane module was implemented with 0.040 inch o.d. Teflon tubing. The other end of the microtrap was connected with 0.165 inch o.d. Teflon tubing. The arrangement of the tubing connected with the microtrap prevented heat loss, back-flushing, and possible short circuits with other parts of this system during desorption. The microtrap was then placed inside the control unit.

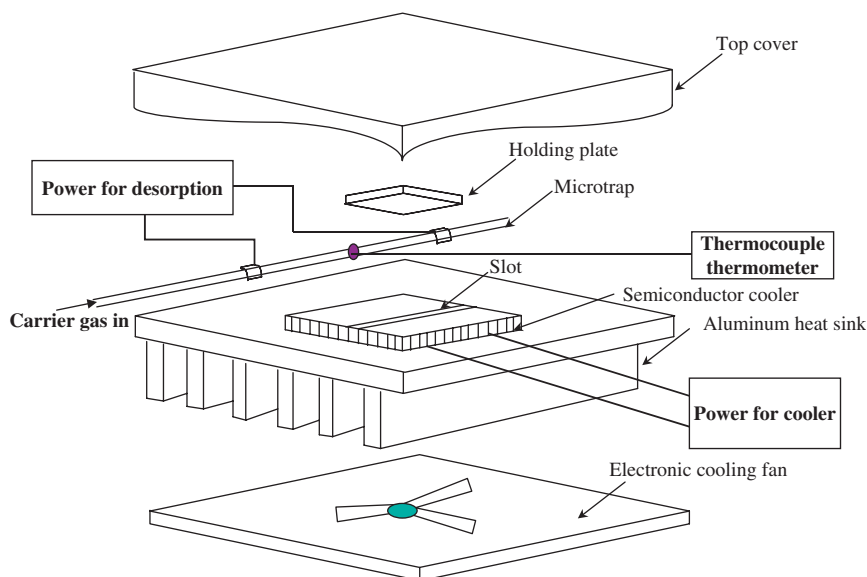


Figure 2. Schematic diagram of the MESI control unit.

A control unit (figure 2) was designed to facilitate temperature control of the microtrap. It consisted of a cooler and a heater unit. The cooling unit was composed of a one-stage peltier cooler and a cooling fan. An aluminium plate with a slot through the centre of the plate was used to cover and fasten the microtrap. Stainless steel, with glass wool packings inside, was used to cover the cooling part to provide insulation. An adjustable 12 V DC power was used to power the cooler. A pulsed capacitor power supply (PS 96, Electronic Shop, University of Waterloo) or a 20 V, 10 A DC power supply was used to heat the microtrap for desorption. The heating current to the microtrap could be turned on for a given duration and at a fixed interval of time. A Fluke 53II thermocouple thermometer (Fluke Corp., Everett, WA), with a sensor wire tightly wrapped around the microtrap, was used to monitor the change in microtrap temperature while applying heating current to the microtrap. A portable SRI GC (Model 8610) equipped with a FID detector, a dry electrolytic conductivity detector (DELCD) [12], a PID detector, and a mega-bore capillary column was selected for use in the on-site analysis, in conjunction with the MESI system. Hydrogen produced by an attached hydrogen gas generator, and regulated by a flow controller, flowed through the transfer line and the interior of the membrane, to serve as either a carrier gas or combustion gas (e.g., for FID). A desktop computer and Peaksimple software (for windows 3.1 version) were used to control the overall operation of the GC system and process the results.

The analytical procedure consisted of two cycles including preconcentration and desorption. In the first cycle, analytes that permeated through the membrane were stripped and then transported by carrier gas to the microtrap, where they were retained until a sufficient amount had been concentrated for thermal desorption and analysis. During this cycle, the microtrap was kept at room temperature, or a considerably lower temperature, with the use of a cooler. The desired preconcentration time was selected to accumulate considerable amount of analytes, which was within the linear

range of the detector and did not result in breakthrough in the microtrap. During the desorption cycle, the microtrap was electronically heated at predetermined times by applying a voltage pulse from the aforementioned power supply. A narrow plug of the analytes was injected into the top of the separation column mounted in the portable GC.

### 3. Results and discussion

#### 3.1. Selections of membrane and microtrap

The selection of a suitable membrane and microtrap was critical to the study because the extraction efficiency of this system depends on the permeation rate through the membrane and the capability of the microtrap to trap the analytes. A PDMS (polydimethylsiloxane) nonporous membrane was selected because it offered a number of advantages. First, it provides rapid transport of analytes due to the high rate of diffusion [14]. Second, it is non-polar, thus preventing water and other polar matrices from entering the system. The Tenax trap was primarily used in these experiments, due to the obvious advantages of Tenax. It has very low background emissions, is easy to clean after sampling, has a low affinity for water, and is thermally stable [15]. However, the concentration of low-boiling-point analytes may be underestimated because of early breakthrough when using a Tenax trap. So, the Carboxen trap, whose small pore size provides the best trapping ability for volatile organics, was used to sample the chloroform in these experiments [16].

#### 3.2. Performance of the monitoring system

The operation of this system was demonstrated by continuously monitoring a standard gas mixture, which was prepared by the standard gas generator. In this study, it contained 8.59 ppm of toluene, 1.12 ppm of ethylbenzene, and 1.96 ppm of *o*-xylene. A linear relationship between system response and sampling time is presented in figure 3. The regression data were determined for three compounds ( $y = ax + b$ , where  $y$  = response intensity in arbitrary units and  $x$  = sampling time in minutes), including; toluene,  $y = 14.455x - 4.09$ ; ethylbenzene,  $y = 1.725x - 0.078$ ; and *o*-xylene,  $y = 1.396x - 0.040$  (correlation coefficients ( $R^2$ ): 0.999, 0.999, and 0.999, respectively). Detection limits, measured on the basis of  $S/N = 3$ , for toluene, ethylbenzene, and *o*-xylene were 0.6, 0.4, and 0.5 ppb, respectively. In general, low detection limits (ppb) were achieved with this analytical system. However, in situations when a higher specificity is required, the detection limits of target analytes may be optimized by adjusting the preconcentration time. Short-turnaround-time analyses can be obtained for high-concentration applications, while for low-concentration samples the sampling time can be extended to maximize the response for the analytes of interest. The reproducibility of the system was tested by on-line monitoring of the above-mentioned standard gas mixture with 15-min sampling times. The measurements were based on six replicate injections. The relative standard deviations for the extracted amounts of toluene, ethylbenzene, and *o*-xylene were 6.3, 5.1, and 2.3%, respectively. A lower RSD value can be expected with a future automated system.

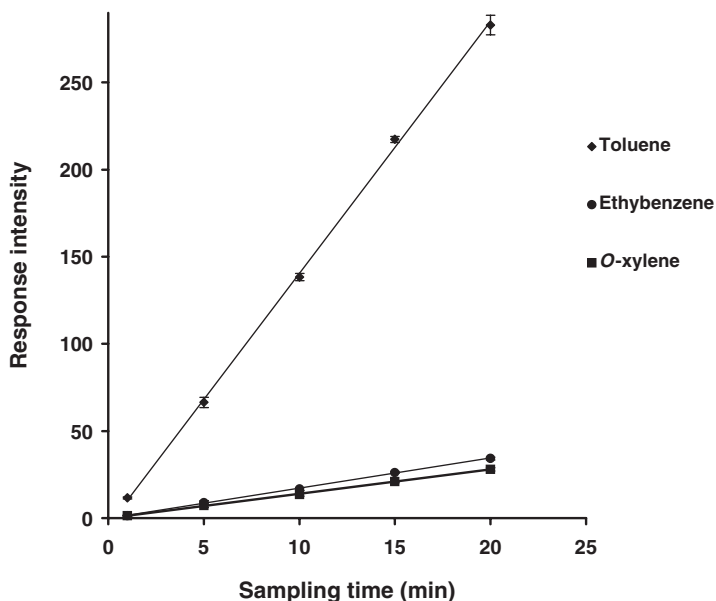


Figure 3. Linear relationship of system response and the sampling time. A Tenax microtrap was used and sampled at room temperature, at a 4.5 mL/min flow rate. An MXT-Volatiles column (10 m  $\times$  0.53 mm, 3.0  $\mu$ m film thickness) (Restek) was used. The analytes were desorbed by using a capacity discharger at 35 V with 1 s pulse width. The PID system was used as the detector. The injector temperature was 200°C, and the detector temperature was 250°C. The initial column temperature was 80°C, held for 10 min, and then increased at a rate of 20°C/min to 180°C.

### 3.3. MESI-portable GC system applications

The MESI-portable GC system can be used for a very broad range of applications in the on-site, continuous monitoring of processes. In order to better explore the features and capabilities of this system, it was tested in various applications, including various air and liquid applications.

**3.3.1. Monitoring of toluene in sewer pipe.** Purifying wastewater with the ultimate aim to provide safe drinking water is a potential alternative to resolve the shortage of pure drinking water around the world. In industrialized nations, including both Europe and the USA, legislation controlling wastewater has become more stringent [17, 18]. These two factors contribute to the importance of on-site monitoring of the quality of wastewater. To determine the application of the monitoring system for wastewater applications, the system was used to monitor toluene in a sewer pipe. This system was transported by a small cart and was installed on-site in half an hour. The sampling device of this system was set up as depicted in figure 4(b). The membrane module was placed inside a tee fitting. The transfer tubings were inserted in two holes in the cap, and a Teflon piece provided a tight seal around the tubings. The membrane module in the designed system was used to simulate a continuous monitoring sensor, and the sealed system prevented pollutants in the wastewater from directly emitting into the air. Figure 5 illustrates on-site monitoring setup at the Department of Chemistry in the University of Waterloo.



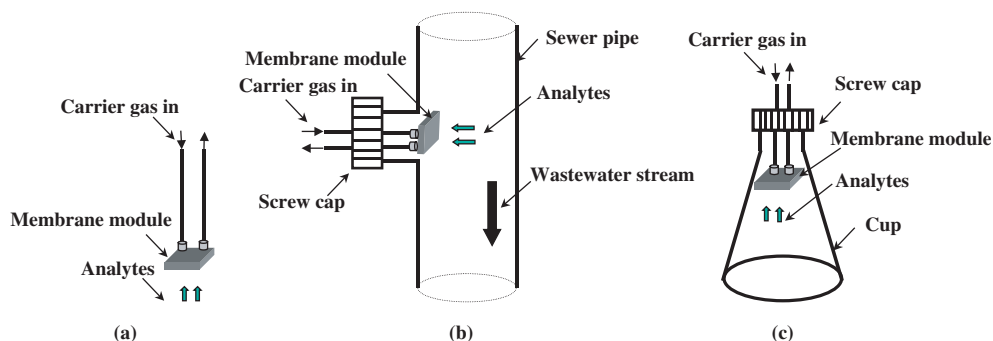


Figure 4. Different configurations of membrane extraction modules for analysis of sample in various sites: (a) fume hood; (b) sewer pipe; (c) swimming pool.



Figure 5. Illustration of toluene monitoring in a sewer pipe at the Department of Chemistry in the University of Waterloo: 1: SRI8610GC; 2: microtrap in control unit; 3: membrane module inside the sewer pipe; 4: power for the cooler (a) and the heater (b).

The results of the toluene analysis are presented in figure 6. The toluene concentration throughout the day varied greatly and reached a maximum level at 3:00 pm. After 5:00 pm, the majority of researchers departed from the site, and the toluene level decreased rapidly. Comparing the static or dynamic headspace gas chromatographic methods (GC or GC-MS), which are the most common methods currently used to analyse VOCs from water samples, it was observed that this system presents a number of advantages, including on-line/on-site analysis, faster responses to the variations of the pollutant concentration, and a decreased matrix effect (especially water-vapour interference). Thus, this system provides a convenient means of on-site monitoring of wastewater.

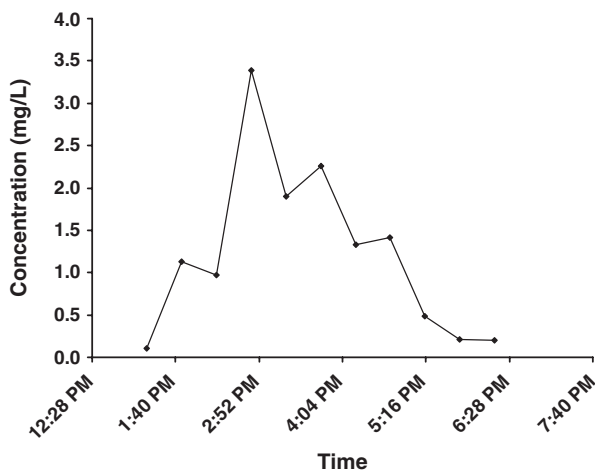


Figure 6. Fluctuation of toluene levels in laboratory wastewater. A Tenax microtrap was used and sampled at room temperature at a 4.0 mL/min flow rate. The sampling time was 30 min. An MXT-Volatiles column (10 m  $\times$  0.53 mm, 3.0  $\mu$ m film thickness) (Restek) was used. The analytes were desorbed by using a capacity discharger at 35 V with 1 s pulse width. A PID system was used as the detector.

**3.3.2. Monitoring air quality of fume hood in laboratory.** Inhalation is a major and relatively unavoidable route of exposure to toxic materials. The analysis of the volatile toxics present in the atmosphere and in the workplace is important to control human exposure to substances that are hazardous to health. For an air matrix, the MESI-portable GC system was investigated as a simple and effective continuous VOC monitoring station. Unlike the traditional methods for air analysis [19], the MESI system eliminates the need for organic-solvent extraction and a drying process, and avoids analyte loss during sample transportation from the field to the lab. This was demonstrated by continuous on-site monitoring of air quality of a fume hood in the laboratory. During the experiment, the membrane module was set up near the centre of the fume hood. The exterior of the membrane was exposed directly to the air while the carrier gas flowed through the centre core of the membrane (figure 4a). Figure 7 shows a set of selected experimental results illustrating the fluctuation in concentration levels of the components in the fume hood at different times during an afternoon. These VOCs were identified by comparing their retention times with those measured in a series of standard sample analyses. The concentrations of benzene, toluene, and ethylbenzene, ubiquitous compounds in a workplace, decreased gradually but did not seem to ever be completely purged. Acetone, the most commonly used laboratory solvent for cleaning GC instruments, exhibited variable intensity, depending on its use. Correspondingly, it can be observed that there was a peak at around 6:00 pm during a sampling day (figure 7) when a person in the lab used acetone to clean the injector. These results clearly demonstrate that the fume hood gradually drew out the pollutants during the day, and the quality of air in the lab improved late at night. Thus, this system is a powerful tool for on-line/on-site fast analysis of the quality of air in a workplace.

**3.3.3. Monitoring concentration of chloroform in the swimming pool.** Among the trihalomethanes (THMs), chloroform is most frequently detected in

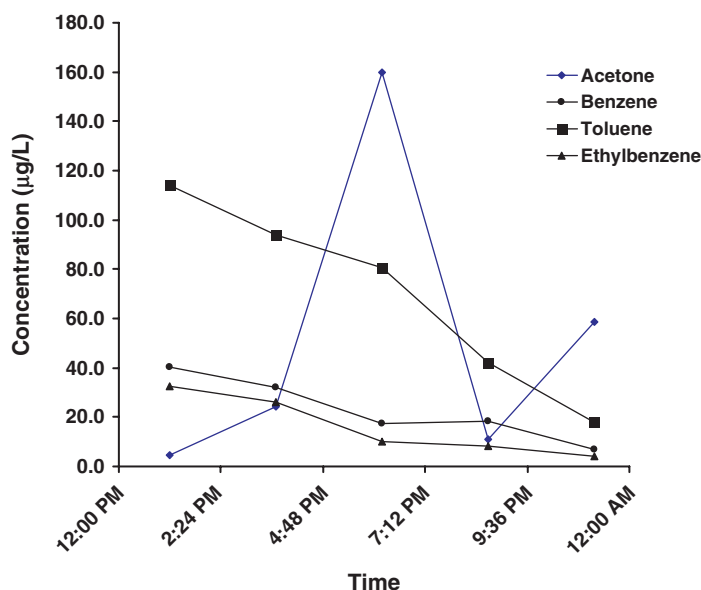


Figure 7. The VOC levels in laboratory air at various times of the day. A Tenax microtrap was used and sampled at room temperature at a 4.0 mL/min flow rate. The sampling time was 30 min. Other parameters were as described in figure 3.

swimming pools [20]. Chloroform has been considered an important factor in a swimmer's health, because it volatilizes easily and may be carcinogenic. Because of its volatilizing capacity, a swimmer can receive a large dose of chloroform through three different routes, including inhalation, dermal absorption, and ingestion. The MESI-portable GC system was used to continuously monitor the fluctuations in chloroform concentrations in a swimming pool. A 'cup' sampling device was set up, as illustrated in figure 4(c). This was a modified conical PET plastic container (210 mm high, diameter of the bottom 80 mm) and was sealed with a cap (diameter 39 mm). The membrane module was held in place in the cone by two transferring tubings, which passed through the two holes in the cap of this cup. This sample system was placed on the top of water steam, thereby creating headspace to facilitate the extraction of analytes. DELCD was used because it is a sensitive detector for halogenated compounds. Two methods were employed to identify the chloroform peak. First, the chloroform was spiked into the water stream, and the suspected chloroform peak was observed to increase significantly. The experiment was repeated several times, using different amounts of chloroform, and the peak of interest increased proportionally each time. Thus, it was concluded that the unknown peak was likely chloroform. Second, the existence of chloroform in swimming pool water was confirmed by SPME-GC-MS experiments. However, the response of the chloroform peak was small. The possible reason was that a considerable amount of chloroform was lost during sample transportation to the laboratory.

A change in chloroform concentration over time was observed in the swimming pool, as illustrated in figure 8. The fluctuation in chloroform levels could be due to a number of factors, including the total organic content in the water, the number of people in the swimming pool, and the water temperature in the

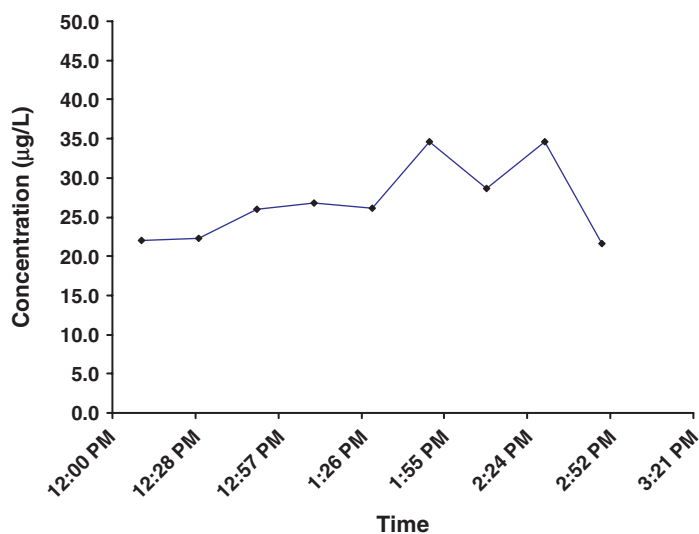


Figure 8. Chloroform level variations in a swimming pool. A carboxen microtrap was used and sampled at  $-5.0^{\circ}\text{C}$  at a  $5.0\text{ mL/min}$  flow rate. The sampling time was 20 min. An MXT-Volatiles column ( $30\text{ m} \times 0.53\text{ mm}$ ,  $1.0\text{ }\mu\text{m}$  film thickness) (Restek) was used. The analytes were desorbed at  $150^{\circ}\text{C}$  using DC power with 0.5 min duration. DELCD system was used as detector.

swimming pool [20]. Moreover, as figure 8 illustrates, the chloroform concentration in the swimming pool was higher (ca.  $25\text{ }\mu\text{g/L}$ ) than that initially anticipated. Most of the health studies of disinfection by-products (DBPs) have been carried out with a focus on drinking water (usually  $10\text{ ng/L}$ ), but swimming pool water seems to present a greater risk of exposure to DBPs (likely due to higher chlorine levels). Therefore, the on-site, real-time monitoring of the fluctuation of chloroform concentration is vital to the process of risk assessment and public-health protection. With these fast and continuous results, suitable responses can be developed. Such measures as adding fresh water, limiting the number of swimming people, and improving the ventilation in the pool can be immediately applied to the swimming pool in order to improve the air quality.

#### 4. Conclusion

The MESI-Portable GC system described herein has been shown to be a very versatile method for continuous, on-site, and reliable environmental monitoring. This system combines sample real-time extraction, preconcentration, and sample introduction in one step, eliminating the steps in sample preparation that are the main source of sample loss and contamination. It can selectively collect analytes according to the quality of the membrane, thus reducing the interferences from the matrix. It is a promising technology for fast, simple, inexpensive, and continuous on-site monitoring, particularly when combined with a miniaturized GC. This system can be applied to other industrial or environmental on-site measurements. New application areas include monitoring emerging contaminants, biological samples, pharmaceutical and cosmetic products, and less volatile and more polar compounds.

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