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Development and Calibration of a Portable Air Sampler

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This paper describes the development and calibration of a portable air sampler for detecting chemical vapors. The air sampler is equipped with a preconcentrator, a battery operated mini-pump, a three-way valve, capacitive sensors housed in a sensing chamber, and a data acquisition and control circuit board. The preconcentrator is used to adsorb trace level chemicals and to thermally desorb them into the sensing chamber. The air sampler was calibrated using known concentrations of ethylbenzene vapor generated by an Environics gas mixing system. The air sampler was also tested using low concentration toluene and ethanol vapors generated by diffusion based vapor generation device. The concentration factor of the preconcentrator was experimentally determined.

1 Introduction

There is significant on-going research in the U.S. and abroad for developing novel sensors for enhanced detection and identification of chemical, biological, and explosives threat. These detectors will need equally efficient front ends for trace level sampling of chemicals. Compared to sensor development, very little research is being conducted on this equally important issue of sampling. The primary objective of our research was to develop an efficient and portable device for vapor sampling, preconcentration and delivery of analytes to novel sensors for detecting trace level chemicals and explosives. Various techniques have been developed in the past for air sampling (1–11). High concentration factors and short thermal desorption time are critical requirements for developing sampling devices for real time detection of trace level chemicals. Most of the air sampling and detection systems currently available make use of commercial preconcentrators that cannot attain concentration factors higher than 100, unless micro-preconcentrator is used (7). In this research, a portable air sampling system was developed in cooperation with Seacoast Science Inc. This air sampler is equipped with a preconcentrator that has a thermal desorption time less than 2 seconds and a concentration factor above 1000.

2 Experimental

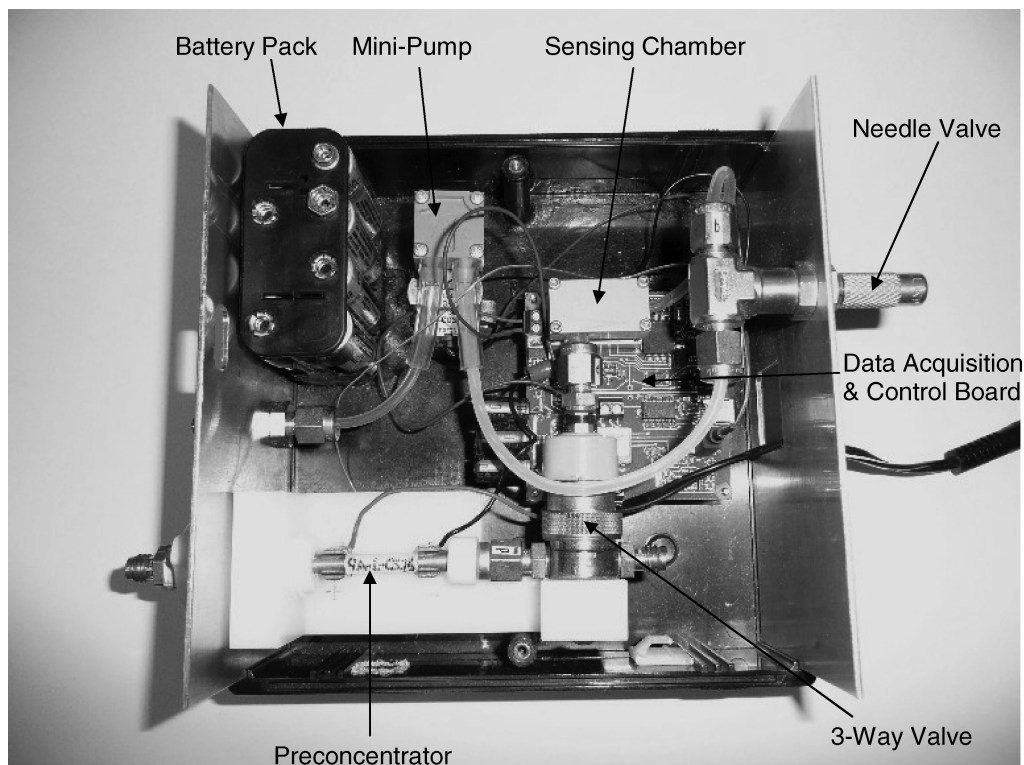
2.1 Description of the Portable Air Sampler

The basic components and schematics of the portable air sampler are shown in Figure 1. It is comprised of a preconcentrator, a pump, a capacitive sensor array housed inside a sensing chamber, a three-way valve, a needle valve, and a data acquisition and control board. The air sampler is connected to a laptop computer via a mini USB cable. The data acquisition and control board is powered by a 7.5V DC power supply made of rechargeable Polymer-Lithium-Ion battery pack. The board also controls the electric current for heating the preconcentrator during desorption phase. The operation of the air sampler, including the pump sampling time, and the thermal desorption is computer controlled. The acquired data is displayed on a computer screen in graphic form in real-time.

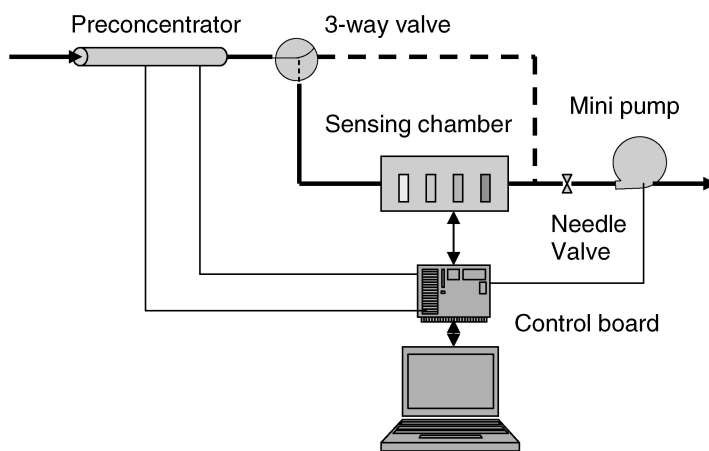
2.2 Polymer Coatings in the Capacitive Sensor Array

The air sampling system detects chemical vapors using a MEMS capacitive sensor array (with eight sensing elements) developed by Seacoast Science Inc. Four different polymers are coated to the four pairs of sensing elements (totally 8 sensing elements), respectively. Figure 2 illustrates the structures of these polymers. Sensors 0 and 1 are coated with polyethyleneimine (PEI), which is shown in Figure 2(a). Sensors 2 and 3 are coated with Bis [(E)-1,1,1-trifluoro-2-(trifluoromethyl)pent-4-en-2-ol] siloxane (ADIOL), which is shown in Figure 2(b). Sensors 4 and 5 are coated with polyepichlorohydrin (PECH), which is shown in Figure 2(c). Sensors 6 and 7 are coated

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(a)



(b)

Fig. 1. (a) The portable air sampler and (b) Schematics of the portable air sampler.

with Polyethylene-co-vinyl acetate (PEVA), which is shown in Figure 2(d). These polymers provide sensitive detection of many volatile organic compounds (VOCs).

2.3 Preconcentration of Analytes and Detection

The preconcentrator is a critical component for trace level detection of analytes when their concentration levels are very low in the surrounding environment. Without preconcentration the amount of material available to detect would be well below the limits of detection for many de-

ectors. The preconcentration method involves passing a high-volume air sample through a tube (preconcentrator) filled with a sorbent material. There are several sorbent materials for the preconcentrator, such as carboxen, carbotrap, carbosieve, activated charcoal, silica gel or Tenax. Two preconcentrator tubes, one packed with tenax and another one packed with carbosieve as the sorbent material, were used in this research. In the sampling phase (sample accumulation phase), the sorbent material adsorbs analytes from the high-volume of airflow. The material is subsequently heated to thermally desorb (release) the trapped analytes

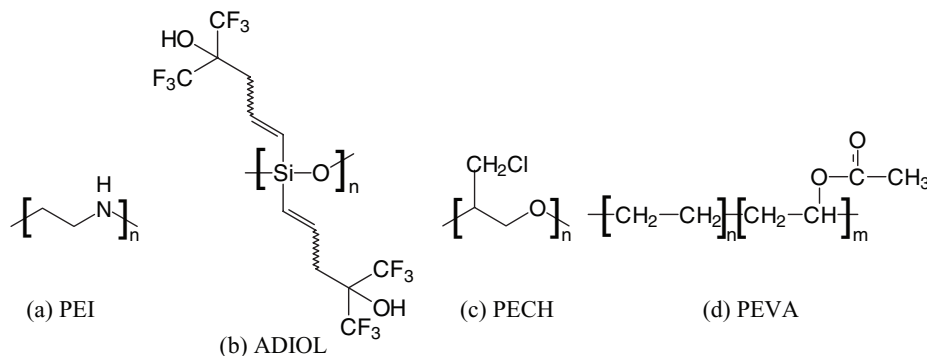


Fig. 2. Molecular structure of the polymers coated on the sensors.

into low-volume airflow stream. Concentration of analytes occur because the samples contained in the large volume prior to the adsorption is now contained (preconcentrated to a higher concentration) in a smaller volume after desorption. The preconcentrated analytes with higher concentration than the sampled vapor is delivered to the sensors housed in sensing chamber for detection. To measure the thermal desorption temperature of preconcentrator, a thermocouple can be inserted into preconcentrator tube. To ensure complete desorption of the absorbed chemicals, the preconcentrator is heated to over 120°C in 1.7 sec (thermal desorption time). To reduce the influence of flow rate on the sensor measurements, the sampled vapors flow through the preconcentrator and the sensing chamber in both the sampling phase and sample delivery phase, as shown in Figure 1(b). The needle valve is used to adjust the sampling rate.

The concentration factor (C_f) of a preconcentrator is defined as the ratio of the amount of adsorbed chemicals in the preconcentrator to the amount of chemicals in the vapor of the same volume as occupied by the adsorbent material in the preconcentrator (8). It depends on both the maximum adsorption capacity of the preconcentrator (which is analyte dependent) and the experimental conditions. The concentration factor (C_f) can be expressed as follows:

$$C_f = \frac{A_d}{\Delta C t_b}, \quad (1)$$

Where A_d is the average peak integrated value, ΔC is the difference of the capacitance readings between the vapor

and the carrier gas, and t_b is the bed residence time expressed as:

$$t_b = \frac{V_b}{Q} \quad (2)$$

Where V_b is the internal volume of the preconcentrator, and Q is the flow rate. For the 0.035 inch in diameter and 0.75 inch length preconcentrator tube, V_b is calculated to be 0.0118 cm^3 . The product of ΔC and t_b represents the amount of chemicals in the vapor occupying the preconcentrator.

2.4 Description of Vapor Generation Systems

The response of the air sampler was tested by two different vapor generating techniques: one is based on a two-stage vapor dilution system (setup by Professor James Whitten's group at UMass Lowell); and the other one is based on diffusion method (12), as illustrated in Figure 3. For the diffusion based vapor generator, the concentration of chemicals is determined by the vapor pressure of analytes at 20°C , the flow rate of the carrier gas, and the dimension of diffusion passage.

The two-stage vapor generation system and the experimental setup for calibrating the portable air sampler is shown in Figure 4. The saturated ethylbenzene vapor was generated by bubbling pure argon through liquid ethylbenzene in a flask. The saturated vapor was then mixed with pure nitrogen gas in an Environics 4000 gas mixing system to obtain the analyte vapor at the desired concentration.

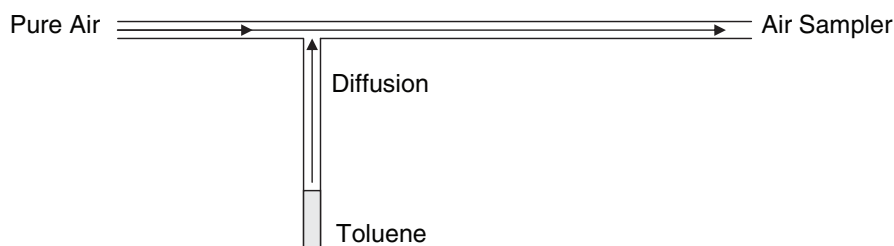


Fig. 3. Schematic of the diffusion vapor generator.

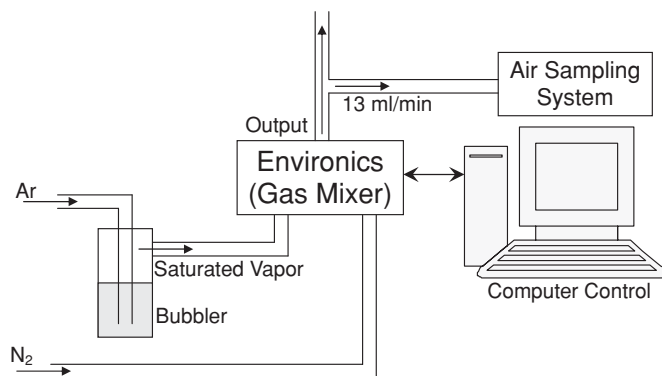


Fig. 4. Vapor generation system and the calibration set up for the air sampler.

The operation of EnviroNics gas mixer is computer controlled. The air sampler was connected to the output port of the gas mixer, and the sampling rate was controlled by the mini pump and the needle valve. In the sampling phase, the ethylbenzene vapor was concentrated by the preconcentrator packed with tenax, and then thermally desorbed from the preconcentrator tube into the sensing chamber (during the delivery phase), where it was detected by the capacitive sensors. For all calibration experiments, the adsorption time was 90 sec and the thermal desorption time (of the preconcentrator) was 1.7 sec.

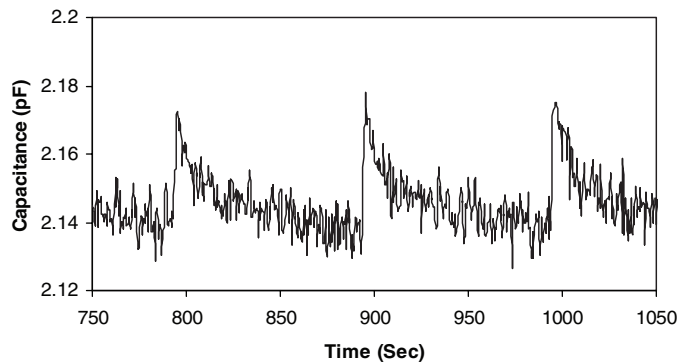
3 Results and Discussion

3.1 Sensor Response to Analyte Vapors

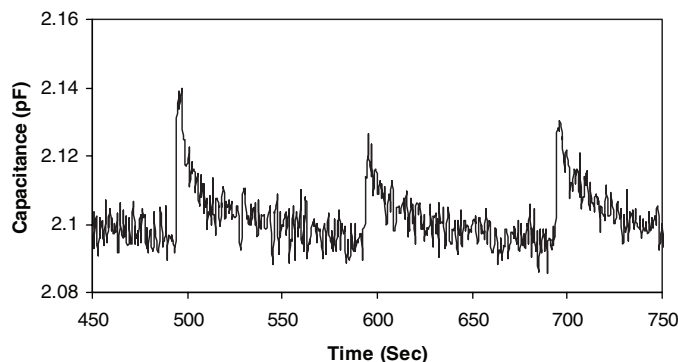
An average response of 0.046 pF was measured for the ethanol/air vapor generated by the diffusion vapor generator, compared to the average response of 0.052 pF obtained from the vapor generated by the two-stage dilution system (Fig. 5). The capacitive sensors are inert to the carrier gases (nitrogen and air). This indicates that the diffusion vapor generator can generate vapor at designed concentrations with relative good accuracy. We also measured the response of toluene vapor at a concentration of 200 ppm using the diffusion vapor generator (Fig. 6). A rapid response from the capacitive sensor demonstrates that the portable air sampler is capable of detecting low concentration chemicals in real time.

3.2 Calibration Based on Peak Integration

The response of sensor 7 was integrated (area under the peaks) and averaged for each concentration. The average peak integrated value is proportional to the amount of chemicals released from the preconcentrator into the sensing chamber. Figure 7 shows the average peak integration as a function of the vapor concentration (from 400 ppm to 2000 ppm). A linear response was observed. This behav-



(a)



(b)

Fig. 5. Sensor 2 response to ethanol vapors generated by (a) Two-stage dilution system and (b) Diffusion based vapor generator.

ior can be expected since for the same thermal desorption time, the peak integration is proportional to the amount of chemicals which is adsorbed and then thermally desorbed from the preconcentrator.

3.3 Concentration Factor of Preconcentrator

To determine the concentration factor of the tenax preconcentrator for toluene vapor, a vapor sample of toluene (277 ppm) was generated by the diffusion vapor generator. The vapor was sent into the portable air sampler at the flow rate of 15 ml/min. To determine ΔC , the tenax preconcentrator was then replaced by a blank tube and the sensor response to toluene vapor at the same concentration (277 ppm) and flow rate ($Q = 15$ ml/min) was obtained. The average peak integration value of 0.7532 pF*sec (A_d) was obtained for sensor 7. The difference of the response to toluene vapor and the response to the carrier gas (air) gave a value of 0.0148 pF (ΔC). The bed residence time t_b was determined to be 0.0473 sec by Equation 2. A concentration factor of 1078 was obtained for the toluene vapor using Equation 1.

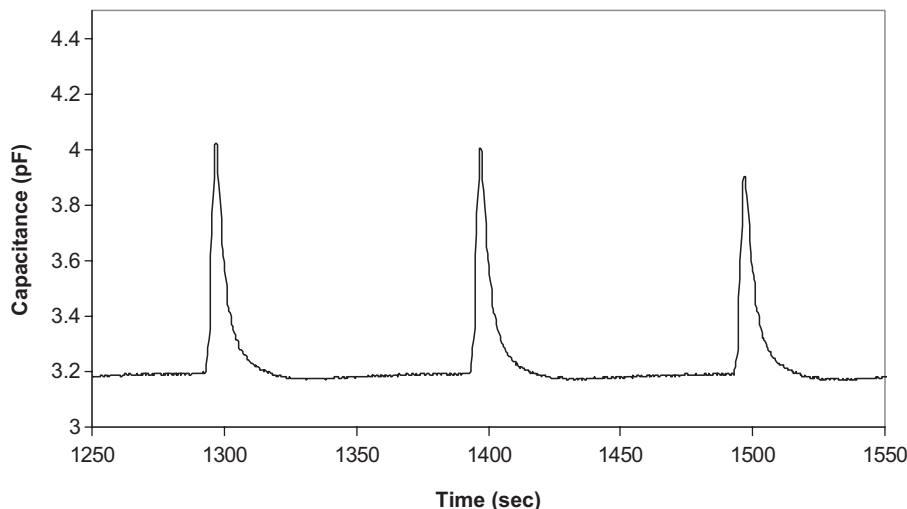


Fig. 6. Sensor 1 response to toluene vapor (22 ml/min, 200 ppm).

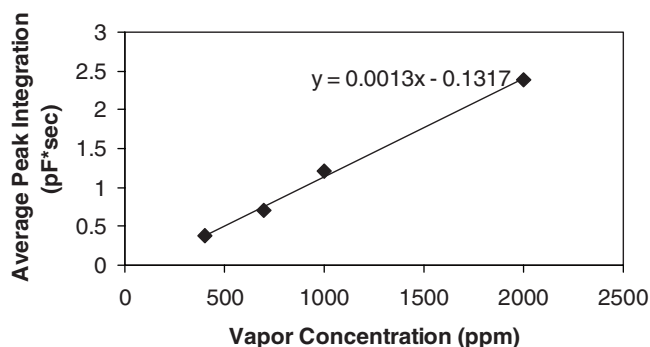


Fig. 7. Calibration of the air sampler for ethylbenzene vapor.

4 Conclusions

A portable air sampler was developed and calibrated. A linear calibration relationship was obtained for ethylbenzene vapors generated by the Environics gas mixer and the bubbler. The air sampler was also tested with ethanol vapor from two different vapor generation systems: a diffusion vapor generator, and a two stage dilution system, respectively. Both vapor generation systems gave consistent sensor responses to ethanol vapors. The concentration factor of the tenax preconcentrator for toluene vapor was found to be over 1000.

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