

A Wireless Hybrid Chemical Sensor for Detection of  
Environmental Volatile Organic Compounds

by

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## ABSTRACT

A wireless hybrid device for detecting volatile organic compounds (VOCs) has been developed. The device combines a highly selective and sensitive tuning-fork based detector with a pre-concentrator and a separation column. The selectivity and sensitivity of the tuning-fork based detector is optimized for discrimination and quantification of benzene, toluene, ethylbenzene, and xylenes (BTEX) via a homemade molecular imprinted polymer, and a specific detection and control circuit. The device is a wireless, portable, battery-powered, and cell-phone operated device. The device has been calibrated and validated in the laboratory and using selected ion flow tube mass spectrometry (SFIT-MS). The capability and robustness are also demonstrated in some field tests. It provides rapid and reliable detection of BTEX in real samples, including challenging high concentrations of interferents, and it is suitable for occupational, environmental health and epidemiological applications.

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## CHAPTER 1

### INTRODUCTION

#### *1.1 Overview*

Human exposure to toxic chemical has become an important problem of public health. Many of these toxic chemicals are volatile organic compounds (VOCs). A group of VOCs include benzene, toluene, ethylbenzene and xylenes, collectively known as BTEX, which are of great concern of many epidemiologists. The main source of BTEX emission (more than 80 %) are automobile exhaust and other traffic related sources [3, 9, 11], indoor sources consist of commonly used paint, detergent, nail polish. Typical BTEX concentrations range from low parts per billion (ppb) by volume [3, 10] to low parts per million (ppm) by volume levels [11]. These VOCs are defined as class A pollutants by US Environmental Protection Agency (EPA) because they are potential carcinogens (US EPA), and many cause leukemia, lymphomas and other diseases [1, 2, 4-9]. Therefore, detection and quantification of trace level BTEX in the air can be critical for many applications, including occupational health, industrial safety, epidemiological study and environmental monitoring. Up to date, gas chromatography and mass spectrometry (GC/MS) is the main technology of VOCs measurement in the air. The sample collection site and the analysis laboratory are usually at different locations, thus sampling collection using canister, teldar bag and adsorbent tube are involved in almost all the GC/MS related VOCs measurement, and are at risk for sample contamination and

loss during storage and transportation. The high cost and long turnaround time limit the data collection in field investigations.

### *1.2 Summary of the following chapters*

The next chapter begins with a brief background of gas chromatography / Mass spectrometry (GC/MS) method and an introduction to quartz tuning forks and their use as sensors, some research work regarding building a portable GC gas detector is also discussed. This is followed by a discussion of the development of a hybrid sensor system that is the focus of this work. The analytical characterization and validation is presented and some field tests results are demonstrated. The final chapters present the author's conclusions and the possibility of future work.



## CHAPTER 2

### BACKGROUND

#### *2.1 Gas chromatography and mass spectrometry*

As we mentioned above, GC/MS is up to date the most common way of analyzing VOCs in a complex mixture. Gas chromatography is a common separation technology used in analytic chemistry. In gas chromatography, a mixture sample with different compounds is injected to a column, the mobile phase is the carries gas, and the stationary phase is a thin layer coating inside the capillary column. As the mobile phase moves along the stationary phase, different compound will be having different interaction with the stationary phase. Depend on how strong the interaction is, the compounds will move in the column at a different speed, result in a different retention time (the time a compound take between the injection and elution), which can be used to characterize the compound. Gas chromatography is usually coupled with mass spectrometry (MS). When a sample is loaded into the MS instrument, the compounds are ionized and then separated according to their mass-to-charge ratio in electromagnetic field. The ions then are detected and a mass spectrum is made from the signals.

#### *2.2 Portable chemical sensor for VOCs analysis*

While reliable, Conventional GC/MS measurement is expensive, not real-time, and often required a trained technician. The tasks of analyzing VOCs in a complex mixture of many interferences with a high sensitivity and selectivity inexpensive portable chemical sensor in real time, is still a challenge. There are

two common approaches for developing a chemical sensor which can detect target analyte in a complex mixture. One common approach of target detection with the present of interferences makes the use of the specific binding between a probe and a target molecule. A strong binding of a probe and a molecule usually results in a high sensitivity, but also means a slow response time and low recovery rate, which requires long sampling time and frequently replacing the sensor, thus not a good candidate for real time detection. Another strategy is gas chromatography technology; a widely used technique is gas chromatography/mass spectrometry (GC/MS). Measurement of VOCs with GC/MS involves the sample collection using canister, teldar bag and adsorbent tube, the detection is not real-time and the cost is substantial. And obviously a bulky and expensive instrument is not suitable for real-time field testing. Commercial portable GC systems have been developed and marketed [12]-[16], but their sensitivity is limited. Carrier gas and computers are needed in some systems and the cost is still high. A miniaturized GC system consists of capillary column, a microfabricated preconcentrator and chemiresistor or surface acoustic wave (SAW) detectors has been reported [17], [18]; The combination of microfabricated GC column and metal oxide(MOX) gas sensors has also been developed [19], [20].

### *2.3 Quartz crystal tuning fork as chemical sensor*

Microfabricated quartz crystal tuning fork have been using in many applications which require precise timing, including wristwatch and microcontroller integrated circuit. Quartz is a piezoelectric material which means

application of mechanical stress results in generation of an electrical signal and vice-versa, and that's the basic principle used in the operation of the quartz tuning fork. The commercial quartz tuning fork are built in a way that the fragile prongs are protected in a metal can; this can also protect the prongs from outside pressure variation and other potential disturbances. Mechanical resonations are generated when a voltage is applied the prongs. The tuning forks require very low power consumption and are very stable and precise.

An important feature of the tuning fork is the high quality factor Q (ratio of energy stored to energy dissipated per oscillation), and this make it suitable for frequency-based measurements. Resonant frequency of a tuning fork can be expressed as follows:

$$f_0 = \frac{1}{2\pi} \sqrt{\frac{k'}{M}}$$

Where  $k$  represents effective spring constant and  $m$  is mass of the tuning fork. The typical Q factor of a tuning fork is 10,000 in the air. In order to use tuning fork as chemical sensor, it need to be modified. We removed the metal can enclosing the prongs, exposing the prongs of the tuning fork. Then, the tuning fork prongs are modified to be hydrophobic, and coated with molecular imprinted polymer. The molecular imprinted polymer forms a thin layer on the prongs, and binds with the target analyte. Once the analyte is bound with the polymer on the prongs, the mass of the prongs increase and therefore decrease the resonant frequency. The change of the frequency is given by:

$$\Delta f(k', M) = \left(\frac{f_0}{2}\right) \left[\frac{\delta k'}{k'} - \frac{\delta M}{M}\right]$$

## CHAPTER 3

### A WIRELESS HYBRID CHEMICAL SENSOR FOR DETECTION OF ENVIRONMENTAL VOLATILE ORGANIC COMPOUNDS

To overcome the difficulties of the current detection technologies, we have recently demonstrated a hybrid approach that integrates specific binding (e.g., colorimetry) and selective separation (e.g. GC) of analytes [21]. The work confirmed the value of the hybrid approach, however, the sensitivity and selectivity fall short to meet the needs of environmental monitoring. In the present work, we introduced adsorbent packed preconcentrator that selectively collects and release analytes, a more sensitive microfabricated tuning fork sensor, an automated heat and flow control with a microcontroller-based circuit, and wireless communication with a cell phone. We have also integrated all the components into a single unit that weighs about 1.2 lbs, tested its analytical performance, and validated its usability in various real world scenarios. The device can reliably detect a few ppb-level of BTEX in complex real samples within minutes, more than three orders of magnitude improvement over the previous work. The system schematic and the performance of the key components of this device are described below.

### *3.1 Experimental setup*

In this hybrid chemical sensor device, three key components are combined: 1) specific pre-concentration for sample collection, 2) chromatographic separation for discrimination of interferents and target analytes, and 3) specific binding for detection of analytes. A distinctive feature of the device is a highly selective tuning-fork based detector. The portable hybrid device can detect VOCs in real-time at few part per billion-levels. While the new detector allows sensitivity, specificity and low size, the preconcentration and separation further improves the selectivity in complex analysis environments. In addition, the device is rechargeable battery-operated and paired to a Smartphone App via a wireless connection, which further reduces weight burden and size. A built-in Bluetooth chip in the hybrid device enables connectivity with a Bluetooth in the cell phone, which not only allows data retrieval from the device to the cell phone, but also remote control of the device from the cell phone. To introduce the working principle of the chemical sensor, we first present the key components, and then briefly describe the measurement procedure.

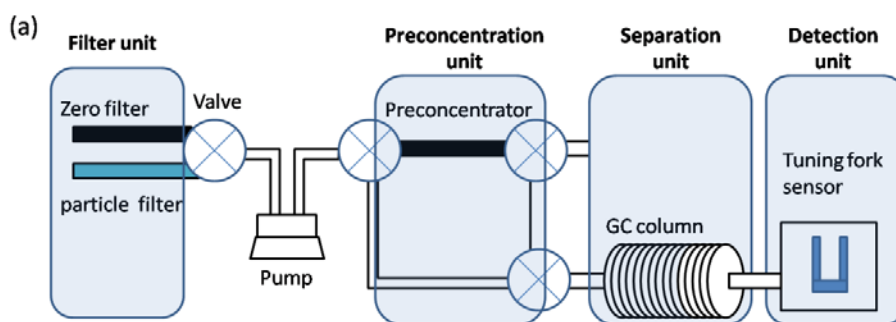


Fig. 1. (a) Schematic representation of the hybrid device. (b) The picture of the hybrid device, insert: the smartphone user interface showing a real time detection of a BTEX mixture sample.

#### A. The Quartz Crystal Tuning Fork Sensor

Quartz crystal tuning forks are mechanical resonators which have a resonance frequency of 32.768 KHz (Newark Electronics) with high quality factor (about 10000 in ambient air) and high sensitivity of mass detection (4pg/mm<sup>2</sup>)

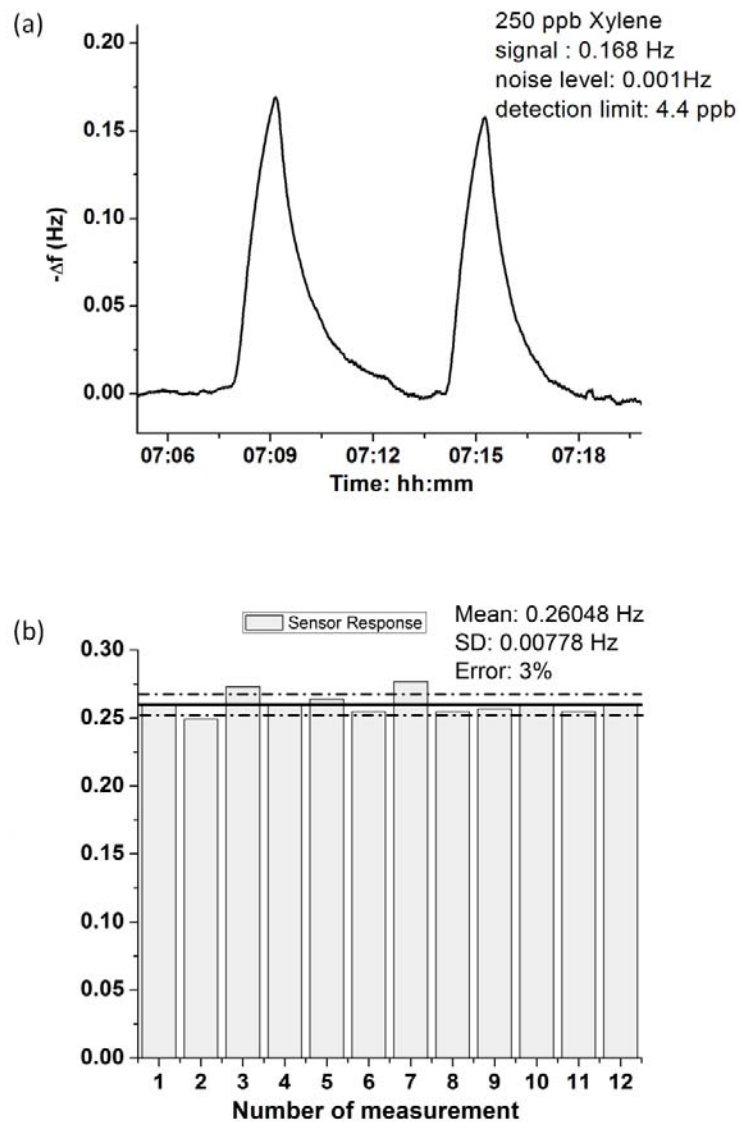


Fig. 2. (a) Response of tuning fork sensor to 250 ppb xylenes. Detection limit: 4.4 ppb xylenes. (b) Results of 12 measurements of the same xylenes sample using the tuning fork sensor.

[22]-[25]. They also have low power consumption (1  $\mu$ W maximum) and a small size (0.1 $\times$ 0.5 $\times$ 3 mm<sup>3</sup>). A digital counter build in the circuit is used to measure the

tuning fork frequency change at a 1.8 mHz resolution. To achieve the desired sensitivity and selectivity, the tuning forks are modified with molecular imprinted polymer (MIP). MIP is highly cross-linked polystyrene formed by divinylbenzene, synthesized with biphenyl as template and xylenes as porogens. The MIP binding sites bind with the hydrocarbons mainly through  $\pi$ - $\pi$  interactions and van der Waals interactions, results in a selective and reversible sensing. The tuning fork prongs need to be hydrophobic so that the MIP can effectively stick to the prongs. To make the tuning fork hydrophobic, the prongs are first soaked in the Phenyl trimethoxy Silane, and then in dodecane ethiol. A MIP-coated tuning fork with ppb-level detection limits for common hydrocarbons like xylenes and toluene is typical. Fig. 2(a) shows a tuning fork sensitivity test. In this test, 250 ppb xylene is prepared and injected to the tuning fork sensor cartridge. The sensor signal is 0.168 Hz, while the noise level is as low as 0.001 Hz. The detection limit is then determined by signal at 3 times of the noise level, which is about 4.4 ppb for xylenes. The sensor also shows a rapid response time and fast desorption process, which are very critical for real-time detection. Fig. 2(b) shows a reproducibility test, in which the tuning fork sensor is exposed to the same concentration analyte for 12 complete measurement cycles. The mean of the responses is 0.26048 Hz, while the standard deviation is 0.00778 Hz, which is about 3% error. Overall, the sensor shows a fast response time, rapid desorption process and excellent reproducibility, together with the low power consumption and super small size features, the tuning fork sensor is a suitable for portable chemical sensor and real-time detection. Common interferences including acetone,



ethanol and ammonia that have been tested at high concentrations (42ppm) shows no response on the MIP-coated tuning fork sensor [26]. The tuning fork sensor used in this hybrid chemical sensor device is after the gas chromatography capillary column, plugged into the circuit board. It is protected inside a Teflon chamber with a very small dead volume which is only 12  $\mu\text{L}$ . The plug and play feature of the tuning fork sensor makes it easy to change and maintain.

### *B. The Preconcentrator*

The preconcentrator located before the gas chromatography column, incoming samples will first go through the preconcentrator and get trapped. It is constructed from a stainless steel tube with a size of 1/16 inch diameter and 2.5 cm length. Packed adsorbent is retained with two glass fiber plug. A Nichrome heating wire which has a resistance of 4 ohms is coiled around the stainless steel tube. The adsorbent can absorb the VOCs at room temperature; a voltage is applied to the heating wire to heat the preconcentrator to a desired high temperature (300 °C) during desorption stage and injection stage. The adsorbent material is the key component in the preconcentrator, and a very important property of the adsorbent material is the capacity. We reviewed adsorbent properties and three different materials are tested. These three materials are Carbopack X, Carbopack B and Carboxen 1016 with surface area of 100  $\text{m}^2/\text{g}$ , 240  $\text{m}^2/\text{g}$  and 75  $\text{m}^2/\text{g}$  respectively. Carbopack B and Carbopack X are graphitized carbon blacks; Carbopack B is non-porous while Carbopack X is porous. Carboxen 1016 is carbon molecular sieve which is the carbon skeletal

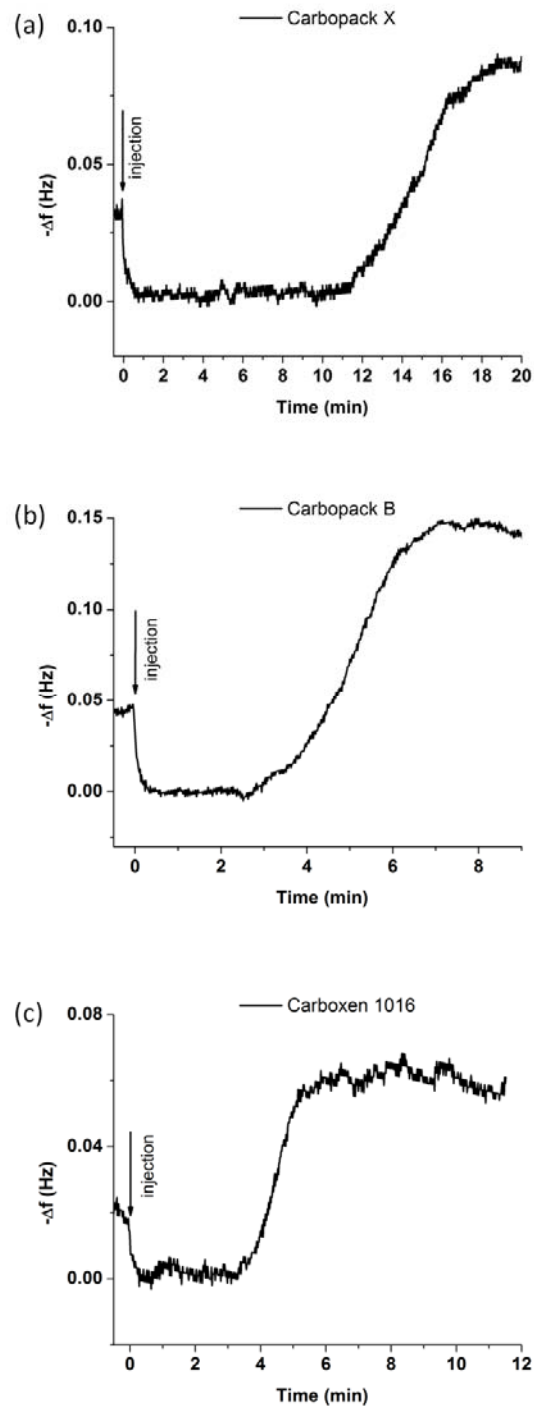


Fig. 3. The breakthrough time tests of three different adsorbents: (a) Carbopack X, (b) Cabopack B, (c) Carboxen 1016.

framework remaining after the pyrolysis of a polymer precursor. A gas sample of toluene at the concentration of 10 ppm is used to compare the breakthrough volume between these three compounds. A miniature pump brings the gas sample which is a 10 ppm toluene into the preconcentrator; a MIP-coated tuning fork sensor with circuits is located at the outlet of the preconcentrator. At room temperature, the adsorbents inside the preconcentrator can absorb the toluene, therefore, as long as the adsorbent materials are not saturated, the toluene can't go through the preconcentrator or reach the tuning fork sensor. Therefore the sensor should not show any positive response of toluene until saturation of the preconcentrator is reached. In the experiments, each material is tested with the same amount which is 12 mg. The flow rate of the toluene gas sample is also the same, which is 700 mL/min. By comparing the time from the point of sample injection to when the sensor starts to show response, we can compare the capacity of each adsorbent material. The responses of the tuning fork for each adsorbent are shown in Fig. 3. It can be observed from Fig. 3 that the breakthrough time are 11.5 minutes, 2.5 minutes and 3 minutes for Carbopack X, Carbopack B and Carboxen 1016, respectively. Carbopack X has longest saturation time, and therefore it has the largest capacity. This is mainly due to the porous property and its high surface area.

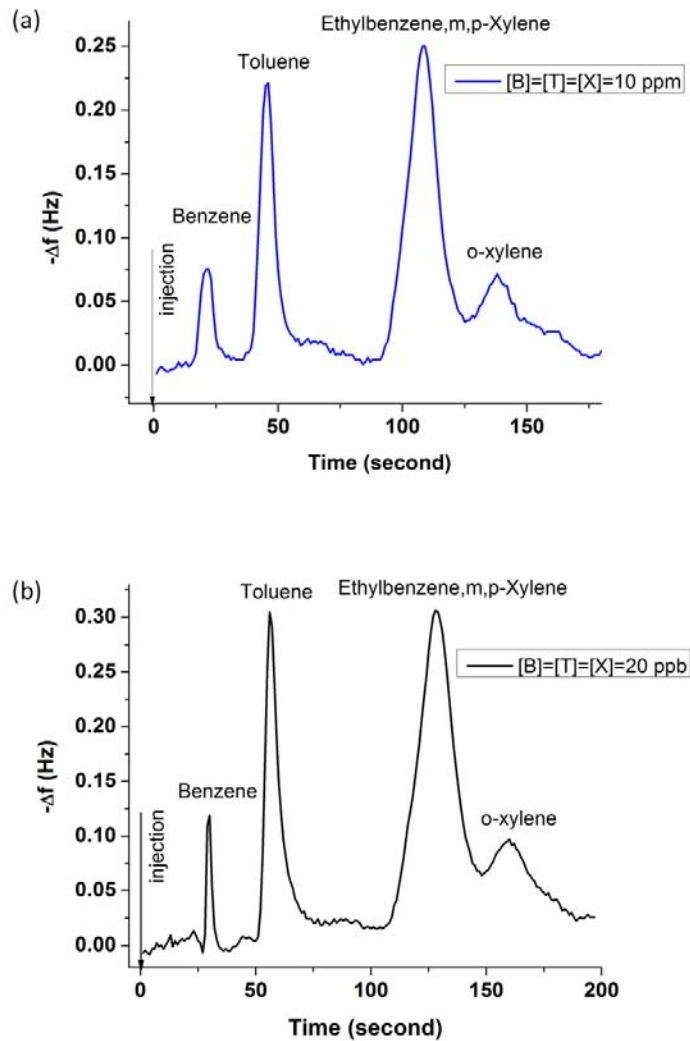


Fig. 4. Chromatograms of BTEX sample with and without pre-concentration.

(a) BTEX sample direct injection without pre-concentration, concentration: 10 ppm. (b) BTEX sample injection with 20 minutes pre-concentration time, concentration: 20 ppb.

An important parameter that describes how efficient the pre-concentrator traps and desorbs the analytes is pre-concentration factor. In our hybrid device,

the factor is defined as the ratio of the tuning fork sensor response of a sample injection using the preconcentrator to that of a direct sample injection without using the preconcentrator. Fig. 4(a) shows the tuning fork sensor response of a direct injection of 10 ppm BTEX sample, and Fig. 4(b) shows the response of a 20 ppb BTEX injection after 20 minutes of pre-concentration. The corresponding pre-concentration factor is ~800 for BTEX. This large preconcentration factor not only reflects the high trap efficiency, but also is a result of high desorption efficiency. We have tested and found that desorption efficient is as high as 99.2%, which is important for repeated detection and analysis of samples. Chemical sensors based on specific molecular binding in general lack this capability for repeated measurements.

### *C. The Gas Chromatography Capillary Column*

The MIP-coated tuning fork sensor provides a selective detection over hydrocarbons family, but including an additional separation mechanism to further improve the selectivity of the system is still desirable. The gas chromatography capillary column located after the preconcentrator and before the tuning fork detector. Two kinds of gas chromatography columns whose stationary phase material are carbowax and cyanopropylphenylsilicone are coupled together to provide the optimal separation. The column with a carbowax stationary phase is UAC-CW (Quadrex) and the column of cyanopropylphenylsilicone is UAC-502 (Quadrex). 2 meters of each column are coupled together, and implemented into the system. The use of the relatively short column provides a fast gas

chromatography measurement while still ensure sufficient separation over BTEX compounds. A typical chromatogram of the BTEX separation corresponding to an injection of subnanomole amount of BTEX mixture for 5 seconds is shown in Fig. 4(b), a complete separation analysis of BTEX only takes 200 seconds, a MIP-coated tuning fork sensor is served as the detector in this test. While providing a fast separation of BTEX, the 4 meters columns sometimes don't have enough capability to separate a very complicated sample. Longer column usually provide a better separation, but the analysis time will also increase significantly. There is a trade-off between the separation efficiency and analysis time, and with the easiness of changing the GC column, different columns can be plugged into this hybrid chemical sensor device to adjust the need of different applications.

#### *D. The circuit*

The microcontroller-based circuit in this chemical sensor device is responsible for controlling, measuring and communication. It times and controls the valve switching between the five different operation stages: preconcentration stage, desorption, injection stage, analysis stage and cleaning stage. It also controls turning on or off the heater. The tuning fork is driven by the circuit, and the frequency is measured. The Bluetooth chip is connected to the microcontrollers so that the command can be sent to the circuit, and the data can be send out to the Smartphone user interface.

### *3.2 Device development, validation and calibration*

#### *A. Development of the prototype of the hybrid device*

The integration of basic components of the hybrid device requires of smart engineering solutions to achieve maximum analytical performance. The analytical performance is evaluated by the capability of the system to separate sample components, and by signal-to-noise ratio quality of each separated component.

Fluids components such as flow rate, dead volumes, materials, and fluid control were optimized and are as follows. First, an optimal flow rate of  $\sim 8$  mL/s is used to reach good sample component separation, avoiding significant diffusional dispersion of sample components inside the column. Flow conditions are reached via a miniature 5.7-V pump (Parker). Second, dead volumes are minimized via home-made connectors to further decrease sample components dispersion, and separation peak broadening. Third, inert materials (Teflon and stainless steel) are used to prevent uncontrolled adsorption of sample components, excepting the preconcentrator, separation column, and detector. In addition, a zeroing filter based on activated carbon (Purafil) is integrated to generate clean air from ambient air as carrier gas, eliminating the burden of using external carrier gas cylinders, and further minimizing weight and size. Lastly, the hybrid device has a fluid system controlled by four miniature valves (Lee Co.) with work in synergic coordination via a built-in control circuit, which is wirelessly activated and set up from the user interface in the cell phone (Fig. 1(b) insert). All above-mentioned components are packed together with the pre-concentrator, separation column, detector, and power components (two lithium-ion polymer batteries) into

a size of 12.9×9.9×4.9 cm<sup>3</sup> project plastic box. The device weights a total of 1.2 lbs. Fig. 1(a) shows a prototype hybrid device along with the Smartphone user interface. The integration of all components leads to a truly portable and self-contained hybrid device for trace level VOCs detection in complex mixtures.

#### *B. The user interface and control of hybrid device function*

The user interface is developed in a Motorola Q phone via Visual Studio (Microsoft) software. The user interface is another key component of the device. It allows receiving, displaying, and storing device data. Once the data have been acquired, it also allows further data transmission via seamless wireless network. As mentioned before it allows controlling the hybrid device functions and its time settings. The device functions consist of five major stages: 1) pre-concentration, 2) desorption, 3) injection, 4) analysis, and 5) cleaning.

1) Pre-concentration stage: In this stage, the pump provides a constant flow of sample (e.g. indoor or outdoor air) to the pre-concentrator, and VOCs are adsorbed. Preconcentration times are adjusted based on VOC concentrations. Preconcentration times are inversely proportional to concentrations. Typically 9-min preconcentration allow detection of few ppb. Once the pre-concentration time is finished, the valves are switched so that scrubbed clean air is purged into the GC column and tuning fork detector registers a baseline.



2) Desorption stage: After the pre-concentration stage, desorption stage starts with the pre-concentration heating to 300°C for 1 min, which allows VOC desorption and release to the gas phase.

3) Injection stage: After desorption stage, the valves are switched again to a injection stage, and scrubbed (clean) air passes through the heated preconcentrator and takes the VOC vapors to the separation column. The injection stage lasts 15 seconds.

4) Analysis stage: After the 15-sec. injection, the analysis stage follows with the separation of sample components in the column via a clean air carrier active flow. Concurrently, the tuning fork detector measures the sensor signals and sends them to the Smartphone. As the sample components exit the separation column, detection id perform from the recorded peaks in a chromatogram (Fig. 4(b)).

5) Cleaning stage: After the analysis time is finished, the device will clean up the pre-concentrator with an additional heating step, and flow of clean air so that the device can be ready for the next testing event.

It is important to mention that the above-described operation is fully automatic, and can be initialed by simply push a button in the Smartphone user interface.

### C. Laboratory Characterization and Calibration

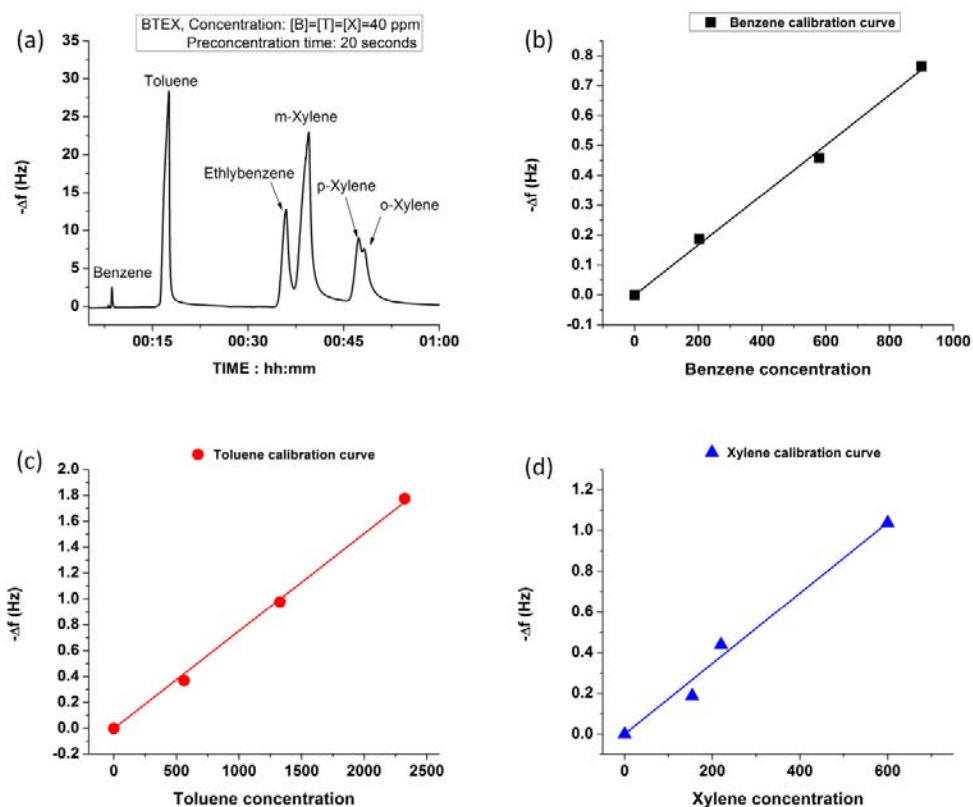


Fig. 5. (a) Separation chromatogram of BTEX mixtures with a 19 meters column [B] is benzene, [T] is toluene, and [X] is Ethylbenzene plus xylenes. (b), (c) and (d) are the calibration curves for benzene, toluene and xylene, respectively.

We have calibrated the hybrid device using BTEX samples with various concentrations. The BTEX samples are prepared and the concentration of each compound is tested with a Selected Ion Flow Tube – Mass Spectrometer (Instrument Science, UK). Fig. 5(a) shows a chromatogram for one of BTEX calibration sample. 19 m column is being used in this calibration test. The BTEX

components elute at the expected elution sequence: benzene, toluene, ethylbenzene and xylenes, consecutively. Also note that the xylene isomers which are m-xylene, p-xylene and o-xylene are also well separated. The high separation efficiency allows the device to analyze a complex sample. The calibration curves (Fig. 5(b-d)) show that the peak heights of each component are proportional to the analyte concentrations. Notice that the linearity concentration goes up to 900 ppb for benzene, 2400 ppb for toluene and 800 ppb for xylene. The sensitivity factors are found to be 0.57 mHz/ppb, 0.50 mHz/ppb and 1.15 mHz/ppb for benzene, toluene and xylene, respectively. The detection limits are a few ppb for all the compounds. It is important to mention that a short pre-concentration time which is 45 seconds is used here; and the detection limits can be further lowered to about 1 ppb by increasing the pre-concentration time to 5 minutes. These large linear calibration range and low detection limit levels are useful for most of outdoor and indoor environmental air monitoring scenarios, as well as industrial applications, and represent an extraordinary good performance for a portable hybrid sensor device. Once the calibration is established, unknown BTEX concentrations can be determined, using either the peak height or peak area of the chromatograms.

### *3.3 Field tests*

In order to test the robustness of the hybrid device in the field, preliminary field tests under different scenarios were carried out and the findings are summarized below.

#### *A. Air quality test during the Gulf oil spill*

The Gulf oil spill in 2011 was a disaster to both the human and the environment. While the seawater was contaminated with over 160 billion of oil gallons, an unknown amount of petroleum VOC contaminated the air of the oil spill area and their surroundings, and represented a high concern hazard for the area inhabitants and workers engaged in oil remediation and cleaning activities [27]. Conventional monitoring equipments showed limited sensitivity and slow response time over this emergency [28]. Taking advantage of the portability of the hybrid device, our team rented a shrimp boat and tested air quality in multiple locations in an area located 69 miles from the spill site (Fig. 7(b)). For this field test, we used a single 2-m column (UAC-502) and 5 minutes preconcentration time.

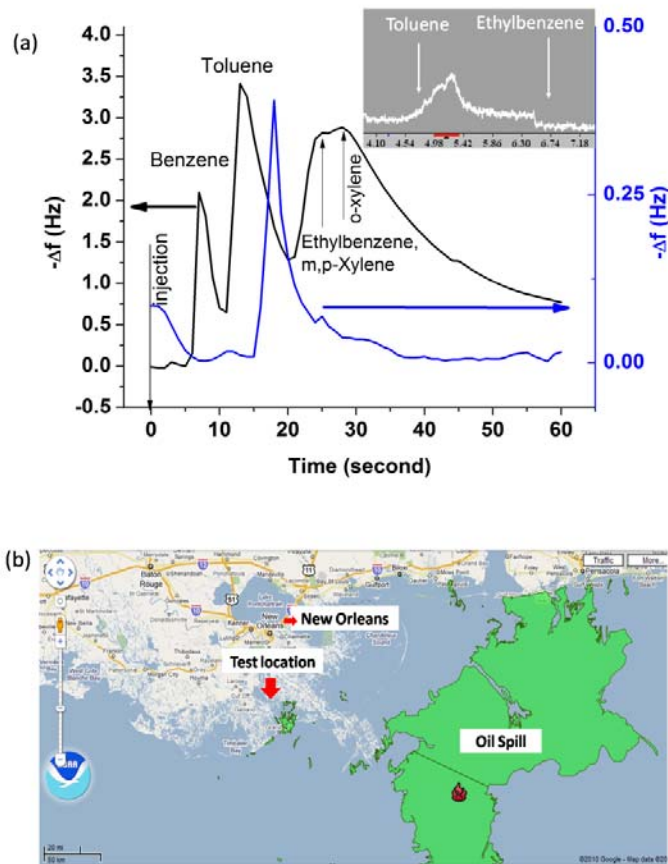


Fig. 6. (a) The testing result of gulf coast (blue line), the typical response of a laboratory prepared BTEX mixture (black line) and GC-MS result (insert).

(b) Map showing test location and oil spill area, date: 6-13-2010.

A typical data set is shown in Fig. 6(a) as a blue curve. For comparison, a 50-ppb BTEX calibration curve (Fig. 6(a) black curve) is also plotted in the same figure. The results reveal two important findings. First, peaks associated with BTEX are absent, which indicate their levels are below 1 ppb. This finding is in agreement with the data reported by EPA' s mobile units, which were deployed in the area at the time of the analysis (June 13th, 2010). The absence of BTEX in

that area is presumably due to the high volatility of the BTEX compounds. Second, a large peak between where toluene and ethylbenzene peaks is found. This peak is due to an alkyl hydrocarbon, and its concentration is estimated to be ~50 ppb. We have confirmed the finding by analyzing water samples collected at the site with GC-MS. The GC-MS data is shown as inset in Fig. 6(a), which reveals a peak between toluene and ethylbenzene. This peak is identified as a hexane derivative, which is in excellent agreement with the finding of our hybrid device.

#### *B. BTEX detection in gasoline vapors*

Gasoline is a petroleum-derived liquid, which is primarily used as engines fuel. It consists mostly of aliphatic and aromatic hydrocarbons. Gasoline vapor is one of the most complicated real samples [29], and is used to challenge the performance of the hybrid device. Because of the complexity of gasoline vapor, a longer separation column is used to provide better separation. Because of the trade-off between the separation and the analysis time, the later is increased to 50 minutes. Fig. 7(a) shows the chromatogram of a real gasoline vapor. The preconcentration time for this sample is 5 minutes, and the analysis time is 40 minutes. About 20 peaks are observed from the chromatogram, which include the

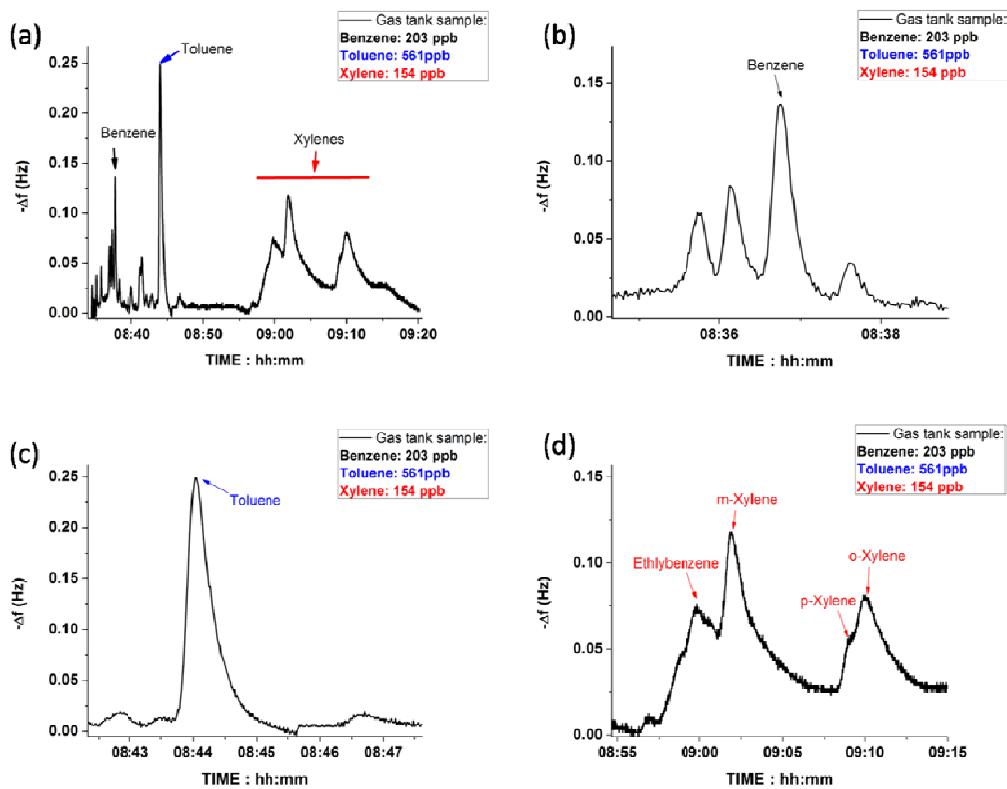


Fig. 7. (a) The test result of a real gasoline vapor and the corresponding detailed separation of the in BTEX compounds: (b) benzene, (c) toluene, and (d) Ethylbenzene and xylenes.

BTEX peaks. Benzene is shown in Fig. 7(b). Fig. 7(c) and Fig. 7(d) present the peaks of toluene and xylenes respectively. Ethylbenzene, m-xylene, p-xylene and o-xylene are also separated in this case. The concentrations of BTEX in this sample are tested using a Selected Ion Flow Tube Mass Spectrometry (Trans Spectra Limited). The ability of the device to detect BTEX in a complex mixture with different kinds of interferents is demonstrated in this test.

## CHAPTER 4

### SUMMARY

A hybrid chemical sensor device has been developed and validated. The key components of this portable device are a mini adsorbent packed preconcentrator, two series-coupled 2 meters or single 19 meters gas chromatography capillary column, and a novel tuning fork detector coated with molecular imprinted polymer. Filtered ambient air is used as the carrier gas. Other features include Lithium-ion polymer battery power source, a Bluetooth communication chip for remote control of the device and the data transmission, and an application in Smartphone for remote command sending, data storage, data analysis and figure plotting.

In most of the interesting applications, including indoor and outdoor air quality monitoring, limits of detection for VOCs in the parts per billion range are needed. The use of the adsorbent packed preconcentrator highly improves the sensitivity of the device. The GC column provide the device a chromatography separation, while maintain a fast measurement process. The use of the tuning fork detector is the most unique part of this device, this tuning fork sensor not only provide a sensitive but also a selective detection of VOCs. The device has been calibrated and validated in the lab with different column length configurations. The device is portable, battery operated, and wirelessly connected to a user-friendly cell phone application. Device applications in real world, such as outdoor air quality and BTEX in gasoline vapor detection are demonstrated.



## CHAPTER 5

### FUTURE WORK

Even though the device has demonstrated its capability, there are improvements need to be done. One concern of the device is the relative long analysis time when equipped a long column. There are several approaches which could address this problem, one possible way is to use a more powerful pump which can provide a higher flow rate; another way is to heat the column. Both will result in an increase of power consumption. Finding a better stationary material to increase the separation efficiency, or using multiple columns in parallel to reduce the pump load and increase the separation efficiency could be solutions, too. MEMS technology could be used to approach these proposals. A microfabricated preconcentrator could be used to reduce the thermal mass and therefore reduce the heater power consumption. Instead of using a plastic box, we could design and machine a box which will be more specific to fit the device components, therefore decrease the device size and increase the robustness. To summary, future work will aim on making the device more selective, sensitive, and faster and smaller, to provide a really valuable tool for various air quality monitoring applications.

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