

Integration of a Chemical Sensor and a Particle Detector

in a Single Portable System

by

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## ABSTRACT

This work demonstrates the integration of a wearable particulate detector and a wireless chemical sensor into a single portable system. The detection philosophy of the chemical sensor is based on highly selective and sensitive microfabricated quartz tuning fork arrays and the particle detector detects the particulate level in real-time using a nephelometric (light scattering) approach. The device integration is realized by carefully evaluating the needs of flow rate, power and data collection. Validation test has been carried out in both laboratory and in field trials such as parking structures and highway exits with high and low traffic emissions. The integrated single portable detection system is capable of reducing the burden for a child to carry multiple devices, simplifying the task of researchers to synchronize and analyze data from different sensors, and minimizing the overall weight, size, and cost of the sensor. It also has a cell phone for data analysis, storage, and transmission as a user-friendly interface. As the chemical and particulate levels present important exposure risks that are of high interests to epidemiologists, the integrated device will provide an easier, wearable and cost effective way to monitor it.

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## Chapter 1

### INTRODUCTION

Wireless Sensor is currently one of the most promising technologies in the world, with the potential to change our private and professional life. From auto industry, environmental monitoring to healthcare, it provides valuable data which was not available before [1-3]. In the near future, we may become even more dependent on them to sense what is going on around us. Particularly, mobile health sensors nowadays are drawing dramatic attention from large major companies, such as Google, Qualcomm, etc [4-7]. With the improvements in automation, efficiency and safety of the sensing system, they will make quite unpredictable contribution to our daily healthcare [8].

#### *A. Volatile Organic Compounds*

Volatile Organic Compounds (VOCs) are organic chemicals that have high vapor pressure in typical room conditions [9]. They are released into the environment in large amounts, with high human toxicity, and a tendency to pollute ground water [10]. One common kind of VOCs, BTEX (benzene, toluene, ethylbenzene and xylene), is typically found in petroleum product, such as gasoline and diesel fuel. VOCs are also released in indoor sources like commonly used paint, detergent and nail polish [11]. Epidemiologists have shown their great concerns about VOCs in public health [12, 13] and various VOCs are known to be toxic chemicals. Additionally, VOCs are defined as class A pollutants by US



Environmental Protection Agency (EPA) because they are potential carcinogens, and may cause leukemia, lymphomas and other diseases [12, 14, 15].

### *B. Particulate Matter*

Particulate Matter (PM), also known as particle pollution, is a mixture of extremely small particles and liquid droplets [16]. They may be either directly emitted from the road traffic and re-suspended or formed within the atmosphere by chemical processes [17]. The size of particles is directly linked to their potential for causing health problems. US Environmental Protection Agency (EPA) is concerned about particles that are 10 micrometers in diameter or smaller because those are the particles that generally pass through the throat and nose and enter the lungs [16]. Such fine particles are able to cause exacerbations of lung diseases and increase blood coagulability [18]. There are observed increases in cardiovascular death associated with urban pollution episodes [11].

### *C. Motivation*

Epidemiologists have found that VOCs and PM present important exposure risks to people, especially children and pregnant women [19-21]. It is always desirable if we can monitor both the chemical and particulate levels simultaneously. However, it is quite challenging to ask a child or a pregnant woman to wear multiple devices. First, the total weight, noise, size, etc. of multiple devices can be an unwelcome burden. Second, different devices have different sampling rate, data collection frequencies and format. It may take

substantial training for the child to learn how to operate and maintain the devices, which can be very time consuming. Finally, the total cost of multiple devices can make it hard to realize personal assessments for every participant in the cohort. To solve the above issues, I have made a cost effective integrated device with only half of the total size and weight and a user-friendly interface to monitor both the particulate and chemical levels simultaneously, leveraging the individual successes of a small sized particle detector platform from Research Triangle Institute (RTI) and a wearable chemical sensor from Arizona State University (ASU).

#### *D. Summary of Following Chapters*

The next chapter includes a brief background description of current technology for chemical sensors and particle detectors and their limitations, together with our solutions and challenges. This is followed by a discussion of the methodology used in the development of a portable integrated detecting system which is the focus of this work. Once a basic theoretical foundation has been established, experimental results are presented along with sensor performance analysis. The final chapters present the author's conclusions and the possibility of future work.

## Chapter 2

### BACKGROUND

#### *A. Chemical Sensors*

A chemical sensor is a device that can transform chemical into an analytically useful signal [22]. The chemical information may originate from a chemical reaction of the analyte or a physical property of the system investigated [23-25]. The functioning of the chemical sensors can be based on temperature change, optical absorption or reflection, mechanical resonance and so on [26]. To make the biggest impact, efforts are mainly focused on detecting pollutants in air with large environmental releases and high human toxicity such as CO, NO<sub>2</sub> and especially VOCs, which is defined as class A pollutants by US Environmental Protection Agency (EPA). The current annual market for chemical sensors is estimated to be worth \$300M and the majority of this market is made up of single gas sensing elements that can be plugged into a portable monitor [27]. For the last two decades, significant research efforts have been made to develop portable and cost effective chemical sensors with low power and long lifetime. However, issues of insufficient selectivity and sensitivity hamper the miniaturized sensors for sensing in environments outside the lab [28].

1) Current VOC Sensing Techniques and Limitations: Toxic VOCs is an important problem as they have been widely found in industrial and domestic areas. It has been shown that physical and psychological effects are associated with exposure to small concentrations of VOCs commonly encountered in daily life [29]. Until recently, measurement of VOCs is mainly conducted by

laboratory based instrument analysis, especially GC (gas chromatography). In gas chromatography, the mobile phase is a carrier gas and the stationary phase is a microscopic layer of liquid or polymer on an inert solid support, inside a piece of glass or metal tubing called a column [30]. The gaseous compounds interact with the walls of the column, which is coated with different stationary phases. This causes each compound to delay and exit the column at a different time (retention time). The comparison of retention times gives GC its analytical usefulness. However, GC typically involves sampling gases with sampler and abstracting the gases from the sampler, which cannot provide real time monitoring [31]. Additionally, the measurement operation is a complicated and skilled high cost job. Since concentration and chemical composition of VOCs can easily change in a short time due to fluctuations in temperature, humidity, air flow and absorption, it is necessary to develop a portable device that can conduct continuous on-site direct analysis with simple process and at reasonable cost.

2) Tuning Fork as a Chemical Sensor: Microfabricated quartz tuning fork is an acoustic resonator in the form of a two-pronged fork. It is widely used in musical instruments, electromechanical watches and microcontroller-based integrated circuits (ICs). Due to the piezoelectric properties of quartz, metal electrodes are plated on tuning fork's prongs so that mechanical resonance can be generated when electrical signal is applied [32]. Tuning fork has very high quality factor Q (ratio of energy stored to energy dissipated per oscillation), which contributes to its low power consumption, high frequency stability and precision. The resonant frequency equation can be expressed as follows [11]:

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

Where  $K'$  stands for the effective spring constant and  $m$  represents the mass of the tuning fork. Usually, the fragile prongs of the tuning fork are sealed in a metal can to be protected from external pressure change and other potential disturbances (e.g. particles in the air). But in our VOCs sensor application, the surface of its prongs is exposed to the outside environment and specially treated to obtain hydrophobic property. Also, a kind of molecular imprinted polymer is coated on the prongs, which forms a thin layer that can selectively bind with VOCs. The tuning fork prongs are extremely sensitive to mass change due to the binding with VOCs, and a delta resonance frequency change can be measured consequently. The corresponding equation is given by [33]:

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

With the progress in manufacturing technique using photolithographic procedure, the mass production of quartz tuning forks has been dramatically improved in terms of size and cost [34], which makes it a portable and cost-effective way to perform VOCs measuring.

### *B. Particle Detectors*

A particle detector is an instrument that can detect the information of particles (e.g. the concentration and the size distribution) for analyzing the cleanliness level in the environment. Usually the detecting principle is based on either light

scattering or light obscuration. Particles get sucked in by a fan or a pump to pass through the detection chamber and then get illuminated by a light source (typically a laser). If light scattering is used, the reflection light intensity will get transferred into an electrical signal for further data processing by a photodiode. If light obscuration is used, the loss of light will be measured. The light blocking method is typically used for detecting particles larger than 1 micrometer while the light scattering method is able to detect smaller particles with sensitivity down to 0.05 micrometer [35]. Particle detectors can provide useful information for determining the air quality inside a building (e.g. clean room) and in the ambient air. They can help protect processes that are very sensitive to environmental contamination and prevent exposure to particles that are directly linked to health problems. In our application, we focus more on the particles 2.5 micron or smaller in size. Once inhaled, these particles can affect the heart and lungs and cause potential serious health effects [36]. Therefore, considering our target particles and the integration with a VOC sensor, we need a small light scattering particle detector which has a matching flow rate with the chemical sensor (500ml/min) and long battery life.

## Chapter 3

### METHODOLOGY

#### A. *Sensors*

1) The Particle Detector: the particle detection module I use to integrate with the VOC sensor is the RTI MicroPEM v2.7 Single-Channel Sampler. It is a tiny but versatile new aerosol sensing system with data collections fully managed by on-board programming.



Fig. 1. RTI MicroPEM v2.7 single channel sampler.

For easier integration with the chemical sensor and further data processing, actually I only use the impactor, optical bench and sampling system of MicroPEM particle detector.

Typically, an impactor is made of a number of classification stages consisting of a nozzle and an impaction plate [37, 38]. When an aerosol stream passes

through the nozzle and impinges upon the plate, particles with large enough inertia will impact upon the plate while smaller particles will pass as an aerosol to the next stage [38]. Typical streamlines and particle trajectories of an impactor are shown in Fig. 2. The impactor of MicroPEM (Fig.3) has a cutoff size of 2.5 micrometers, which can make sure we are detecting the particles of interest.

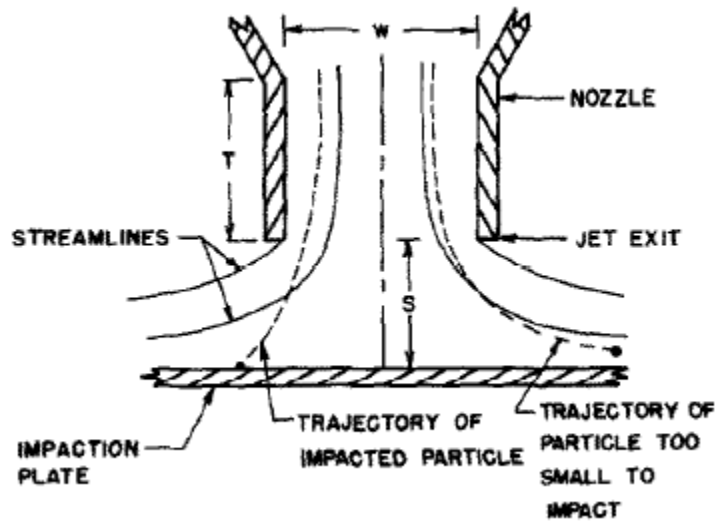


Fig. 2. Streamlines and particle trajectories for a typical impactor [38]



Fig. 3. Impactor components of RTI MicroPEM v2.7 sampler



The optical bench contains a supporting circuit, a laser and a highly sensitive photodiode. Particles get sifted through the impactor and then enter the testing chamber. When particles pass through the laser beam, the scattered light will be sensed by the photodiode and the light intensity change will be transferred into a voltage change. The supporting circuit will amplify the electrical signal and further process it.. With a calibration curve, we can find the corresponding particle concentration. The detecting principle diagram is shown in Fig. 4. As for the sampling system, 500mL/min air flow needs to be guaranteed to ensure the impactor to function. Also, to prevent VOC absorption, the connection tubes have inert Teflon inner layer and all components need to be aligned so we can get a large capture angle of the reflected/scattered light. The efficiency of the optical bench was verified with test dust before integration as shown in Fig. 5.

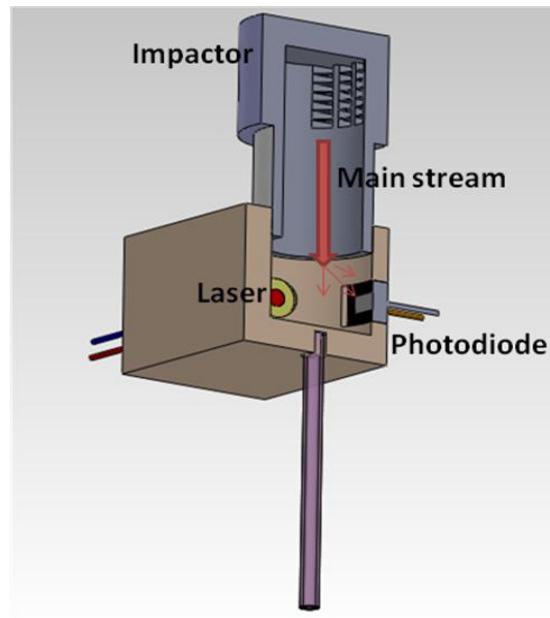


Fig.4. Detecting principle diagram of MicroPEM sampler

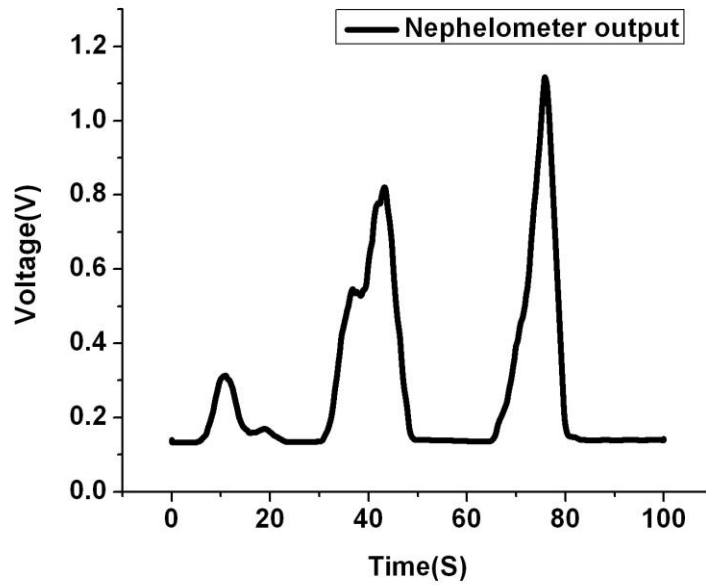


Fig. 5. Verification test of the optical bench with test dust

In the verification test of the optical bench, whenever the inlet of the impactor got exposed to the test dust, a group of peaks would be detected. The noise level is around 0.01mV. So according to accepted general standard, the detection limit is considered to be 3 times the noise level, which is 0.03 mV. According to the calibration curve in Fig. 6, that corresponds to  $0.1\mu\text{g}/\text{m}^3$  in the way I use the optical bench.

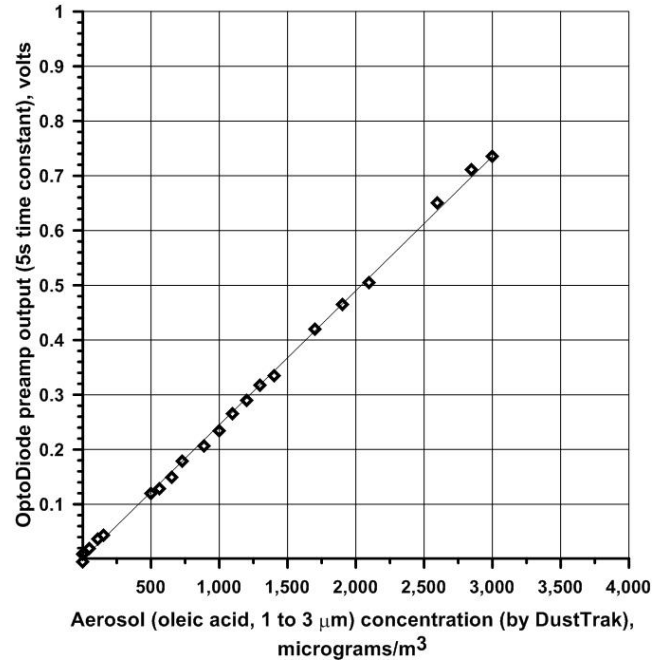


Fig. 6. Calibration curve for the optical output of MicroPEM sampler

2) The VOC Sensor: To realize the sensor integration, I also need to duplicate and improve a wireless VOC sensor developed by ASU, which achieves specific VOC binding with sensitive microfabricated tuning fork sensors, flow control with a microcontroller-based circuit and wireless communication with a cell phone.



Fig.7. The wireless VOC sensor developed by ASU

The VOC sensor has an alternating sampling and purging phases. In sampling phase, the air is sucked in by a pump, filtered by a particle filter and then goes through a nafion tube, which maintains the humidity of the sampling air constant. After that the sample flows into the test cartridge where highly selective and sensitive tuning fork sensors perform real time VOC detection at parts per billion level. The basic working principle is shown in the device schematic in Fig. 8. In the purging phase, the sampled air passes a zeroing filter which filters most of the interfering chemicals and particles so that the tuning fork sensors are blown and cleaned to be ready for the next round of sampling.

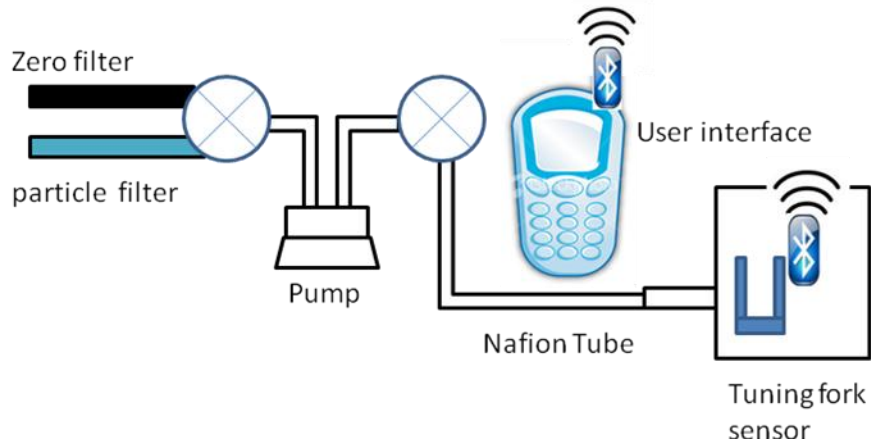


Fig.8. VOC sensor schematic

The device is rechargeable and paired to a cell phone App by a built-in Bluetooth chip, thus allowing data collecting and remote control. Detailed information about the key components and measuring procedure will be discussed in the following section.

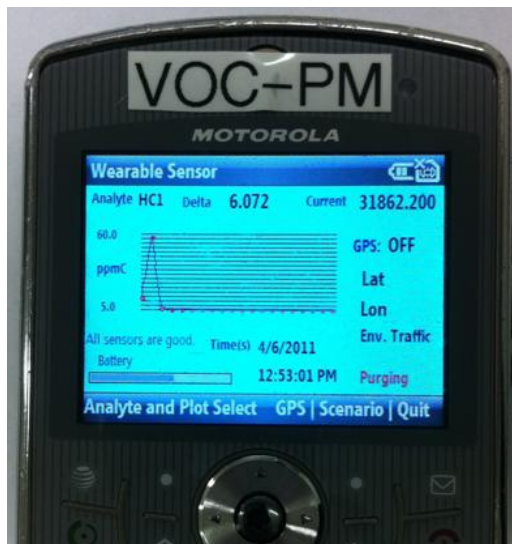


Fig.9. Cell phone user interface showing a real time VOC detection

a. The Sensor Platform: The sensor platform includes quartz tuning fork (QTF) sensors that have a 32.768KHz resonance frequency,  $4\text{pg}/\text{mm}^2$  mass detection sensitivity and can measured 1.8mHz frequency change with a digital counter in the circuit. For high selectivity and sensitivity purpose, the surface of QTFs are chemically treated to be hydrophobic and coated with molecular imprinted polymer (MIP), which can bind with hydrocarbons through  $\pi$ - $\pi$  interactions and van der Waals interactions [11]. This process is completely reversible and results in a typical ppb-level detection limit. Fig. 10 shows the results of a sensitivity test of the tuning fork sensors with 1ppm xylene. The signal to noise ratio is about  $0.485\text{Hz}/0.0008\text{Hz}=606$ . If we take 3 times the noise level as detection limit, it turns out to be 1.65ppb.

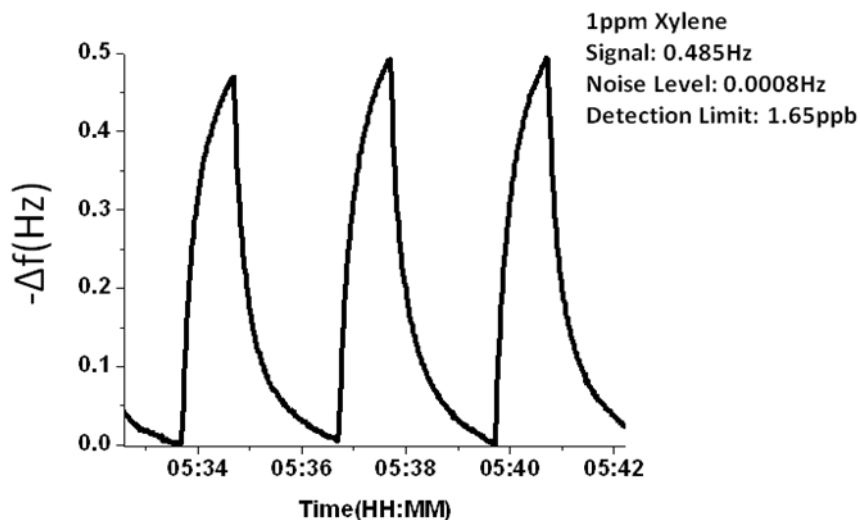


Fig. 10. Tuning fork sensitivity test

b. Filter Subsystem and Sample Delivery System: The filter subsystem is composed of a zeroing filter and a particle filter, which take turns to operate in different modes (sampling and purging ). Its function is mainly to improve the selectivity of the sensing system since in real world testing, we cannot avoid the presence of high concentration interference. Also, it can prevent overexposure and contamination to the QTF polymer coating. The sample delivery system includes a pump, a valve and a microcontroller based circuit. The circuit controls the valve to switch between sampling stage and purging stage, generates electrical signal to drive the QTFs, continuously measures the tuning fork resonance frequency and communicates with a Bluetooth chip for data and command transmission. Additionally, a nafion tube locates before QTF sensors to maintain the humidity of the sampling air constant. Thus, we can get a robust portable device, which can perform in different VOC exposure levels.

### *B. Integration Strategy*

1) Sensor Fusion: After carefully studying and discussing the feasibility of the integrated device and the complementary properties of the two systems, I have come up with a sensor fusion design. The chemical sensor uses filters to remove particles as interference and the particle detector detects the particulate level in real time using a nephelometric approach. By placing the QTF sensor cartridge in the downstream of the particle detector, both the chemical and particulate levels can be determined. A sensor fusion schematic is shown in Fig. 11. Detailed

technical plan including the needs of flow rate, power and data collection will be discussed as follows.

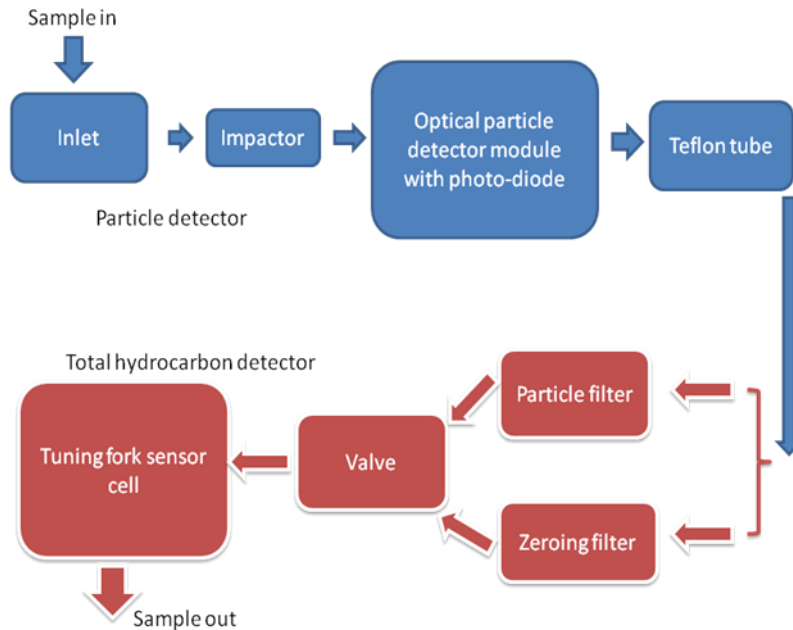


Fig. 11 Schematic of the sensor fusion design

The air sample goes through the particle detector first and then is directed into the inlet of the chemical sensor by a mechanical interface in between which maintains the sample integrity and flow rates requirement (500ml/min for both the devices). A new microcontroller-based Bluetooth enabled circuit is built to read the output signals of both the photodiode of the particle detector and the frequency counter of the chemical sensor. The chemical and particulate exposure levels are transmitted to the existing cell phone platform, which gets modified to include the data processing, storage and display capabilities for both the chemical sensing and particulate detection. The integrated sensor shares the same battery pack, pump and valve so the weight and cost savings can be as much as 50% of



the combined total of the two separate devices. After pre-pilot performance testing, a new housing is developed to encompass all the components of the integrated sensor into a wearable format. Fig. 12 shows how the integrated device looks.

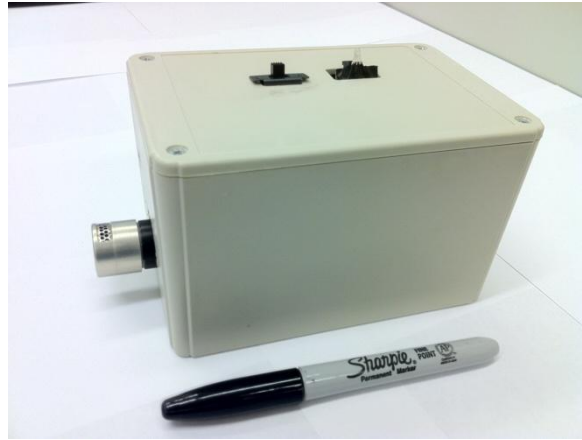


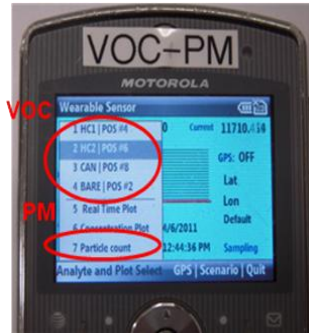
Fig. 12. The appearance of the integrated device

Stats	Wearable unit	User Interface
L x W x H	5.8 in × 3.9 in × 3.3 in	4.6 in × 2.6 in × 0.6 in
Weight	1.6 Lbs	0.34 Lbs

Table 1. The dimension of the integrated device

2) User Interface: Considering that most people including children and adults are already familiar with the operation of cell phone, we develop the user interface in a Motorola Q phone via Visual Studio software, which includes temperature correction, curve fitting and calibration for further data processing. It

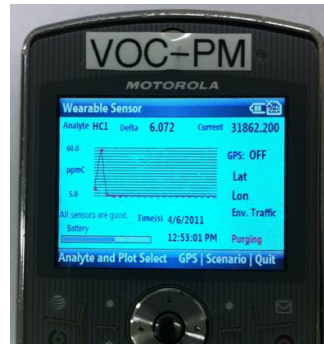
can also store, email and transmit data, either automatically or as needed, to a central location, the epidemiologist's computer or another cell phone. Additionally, it provides the visualization of the measured chemical and particulate exposure levels in a user friendly display (Fig. 13). And the built-in accelerometer and GPS in the cell phone can help enable spatial and temporal mapping of both concentration levels. By simply pushing buttons on the smart phone user interface, full control and operation of the integrated device can be implemented.



(a)



(b)



(c)

Fig. 13. User interface (a) control panel (b) particle display (c) VOC display

## Chapter 4

### RESULTS AND DISCUSSION

Integration of two devices is different from putting them side by side. After integration, two systems share components and start to influence each other. Therefore, the robustness and efficiency of the integrated device need to be tested under different scenarios. Two field tests were carried out and the findings are summarized below.

#### A. Parking Structure

The parking structure 4 in ASU is usually packed after 11 a.m. When the vehicles pass by, VOC and particles will be released from their exhaust tubes. Thus, it is a good location for me to do a verification test for my integrated device.

I started the test at around 8 a.m. and ended it at around noon. My integrated device was set up at a parking spot close to the structure's entrance together with the user interface cell phone.

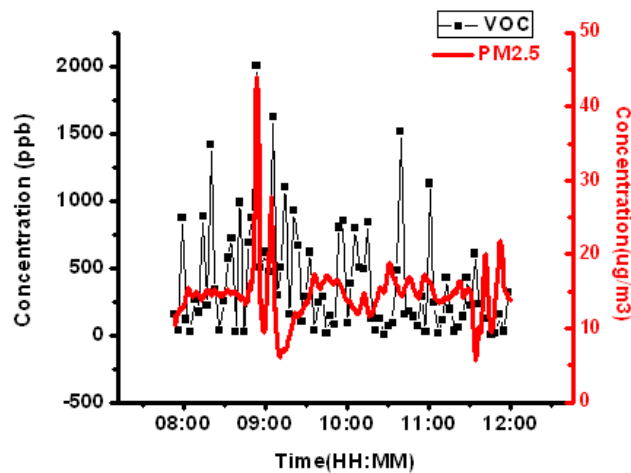


Fig. 14. Test results from parking structure

The test results from the cell phone are shown in Fig. 14. The particulate level is in solid red line while the VOC level is presented by black points and line. The VOC sensor only samples for one minute every three minutes to get one data point since it needs the other two minutes for purging. However, the particle detector collects data every second. To obtain the same resolution, the particle data was averaged for every 180 points. As can be seen, both the PM and VOC have sharp peaks from 8:30 to 9:00 a.m. because that is when most vehicles come in. Otherwise, the PM level is relatively stable until noon when some people go out for lunch. As for the VOC level, there are single peaks every now and then from 10:00 to 11:00 a.m., which may due to exhaust tubes of the cars parking nearby.

The results indicate that in parking structure 4, the particulate level is about  $15\mu\text{g}/\text{m}^3$  and the VOC level is about 500 ppb on average. According to ADEQ daily air quality report [39], the average PM<sub>2.5</sub> concentration around Tempe is about  $3\mu\text{g}/\text{m}^3$ . Since ADEQ does not report VOC concentration on a daily basis, the CO concentration data (300ppb) in the report is used as a VOC concentration indicator, based on the fact that vehicles exhaust both CO and VOC at the same time. The air in the parking structure is expected to be more polluted, so the results are in agreement with what I expect. Also, both the PM and VOC levels show peaks during the rush hour from 8:30 to 9:00 a.m. Thus the ability of the integrated device to test both VOC and particles in real world simultaneously is verified.

### B. ADEQ Greenwood Station

ADEQ Greenwood Station is an ideal location for us to test and correlate our device with reference instruments. First of all, it is located near an exit of Highway I-10. People go to work through I-10 so in the mornings of weekdays, there will be heavy traffic around the station. In addition, the ADEQ can provide daily reports of PM<sub>2.5</sub> concentration and CO concentration. Although the station equipment and our device may not sample exactly the same air and CO concentration may not reflect the total VOC concentration, the trend of the concentration change should roughly follow each other since my integrated device and ADEQ test instruments are basically in the same location.

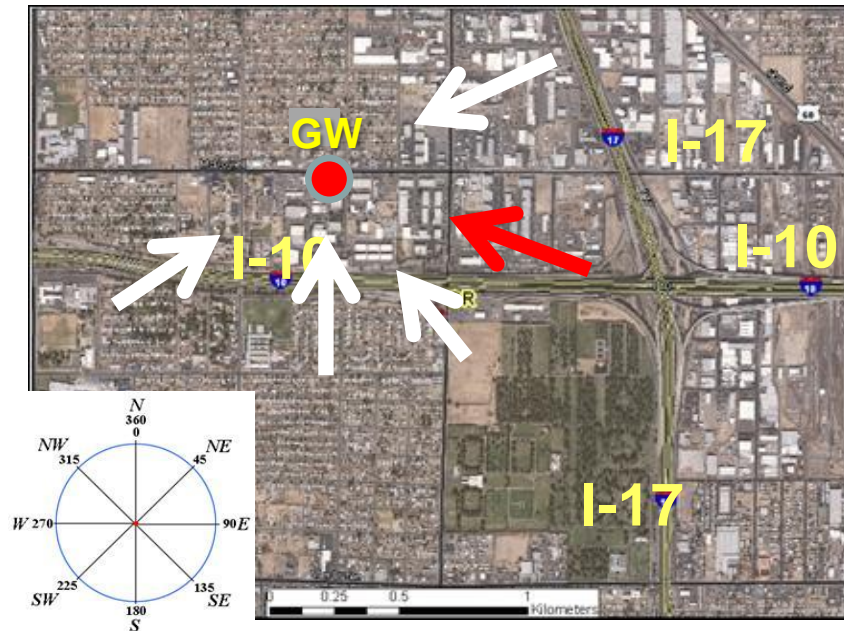


Fig. 15. The ADEQ Greenwood Station location

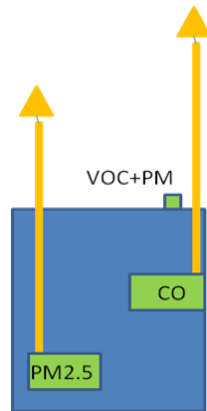
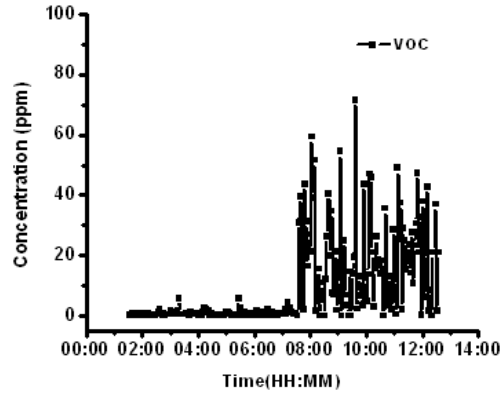
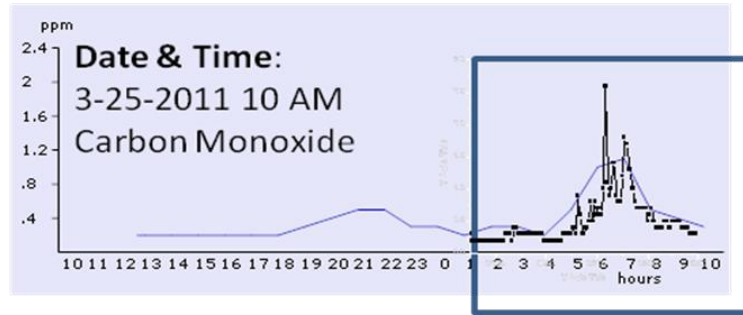


Fig. 16. Location of the reference instruments with respect to the integrated device

The test started at around 1 a.m. and ended at around noon. The integrated device was placed on the roof of the station while the reference instruments were inside the room with the sampled air being drawn from the outside. The ADEQ air quality monitoring data was downloaded from its main website [40] and we also got a copy directly from the station as another reference. The testing results will be discussed below.



(a)



(b)

Fig.17. (a) VOC concentration data from the integrated device (b) CO concentration data from ADEQ [40]

The VOC concentration increase correlated with the traffic activity increase around 6:30 to 7:00 a.m. and during that time, the CO concentration got its peaks as well. Since the traffic can exhaust CO and VOC at the same time, CO is a dominant indicator for VOC. However, after all, CO and VOC are different chemicals, their concentrations differ but a correlation between their concentration changes was observed. The dotted data in Fig. 17 (b) was directly gotten from the Greenwood Station, which was sampled every five minutes.



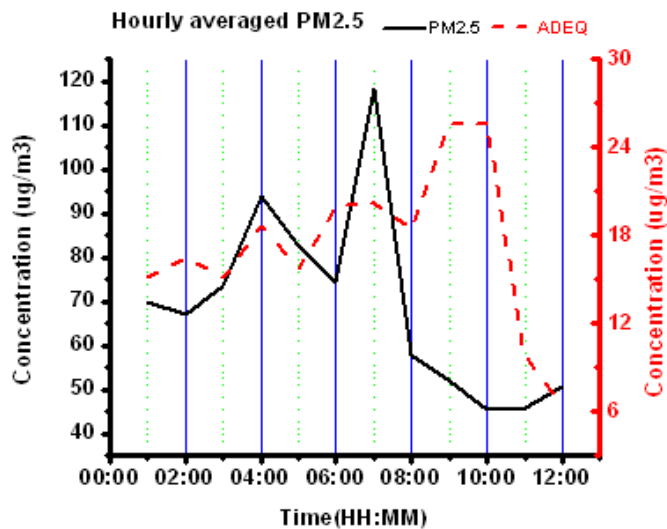


Fig. 18. Particle concentration data from both the integrated device and ADEQ daily report [40]

Since the ADEQ only provides data points for PM2.5 level every hour, the PM data from the integrated device was hourly averaged to be more comparable. The ADEQ data is in dash red line and our data is in solid black line. As shown in Fig. 18, the overall trend follows each other except the peak around 9:30 a.m. in the ADEQ data. Since there is desert surrounding the station so the particle concentration also correlates with the wind speed and direction, which was very unstable. That is why we see ups and downs in the PM data. Around noon, both the integrated device and the reference instrument detected the minimum in particle concentration. Overall, the integrated device showed very good performance and its efficiency is validated.

## Chapter 5

### CONCLUSIONS

This work has demonstrated a new version of an integrated particulate and chemical detection device for children health study with a unified cell phone user interface. The sensor's basic function has been demonstrated in field tests. It has been proved to be able to monitor air quality by selectively detecting ppb-levels of traffic related hydrocarbons and concentration of particulate matters in real time. Also, this reliable multifunctional monitoring device reduces the total cost of devices involved and burden on the subjects including size, weight and operational maintenance. Another important benefit of the combined device is the ability to perform multiple analysis of the same sampled air, which is not usually available to portable devices.

## Chapter 6

### FUTURE WORK

The integrated device has shown good performances in the field tests but it still needs some improvements. First of all, the ultimate goal of the project is to make a completely multifunctional integrated device, including VOC sensor, CO sensor, NO sensor, metabolic sensor and particle detector in a portable format since they are related to different kinds of health problems. This goal can be solved by adding other extra combination of polymer-modified QTFs and interferent filters to interact with different analytes. Actually, some initial work is already in progress.

Another concern is how to make the device even more portable. We can try to design and machine a metal box which specifically fit the device components instead of using a generic plastic box. Thus the robustness and portability of the integrated device will also be improved.

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