SAND2012-1388C

Optimized SAW Chemical Sensor with Microfluidic Packaging

Robert W. Brocato, Terisse A. Brocato, Joel R. Wendt, Carlos A. Sanchez, and Larry G. Stotts

Sandia National Laboratories*, P.O. Box 5800 Albuquerque, NM 87185 USA (phone: 505-844-2714; fax: 505-844-7011; e-mail: rwbroca@ sandia.gov).

Abstract— **Surface acoustic wave (SAW) devices are used as sensing elements in the best performing portable chemical detectors. The SAW device, with a selectively absorbing chemical coating, serves as a mass sensor which preferentially responds to various chemical exposures. To obtain the highest performance, a number of criteria must be optimized, including SAW microwave insertion loss, impedance matching, electrode design configuration, RF shielding, chemically absorbent coating area, electronic measurement approach, and microfluidic packaging. A properly optimized system can be sensitive to chemical exposures the parts-per-trillion range. We report on a design optimization approach consisting of multiple comparison experiments made with competing designs.**

Index Terms—surface acoustic wave, sensors, RF packaging, microfluidic.

I. Introduction

The Microchemlab program was initiated at Sandia National Laboratories in the late 1990's to create a chemical sensor that was highly sensitive to specific chemicals in air, was handheld and portable, was quick, easy to use, and was low cost (fig. 1). The units worked reasonably well at satisfying these widely divergent requirements. The Microchemlab program was based on three key emerging microfabrication technologies that enabled chemical analysis to be performed in a portable, handheld device. These technologies are the micro-machined preconcentrator, the micro-gas chromatograph (GC) column, and the coated, selectively absorbing SAW detector (fig.2).

The preconcentrator is made from chemically selective coatings applied to a micro-machined silicon nitride membrane. The preconcentrator forces incoming sampled air to flow through a tortuous path that is coated with a selectively absorbent, porous sol-gel material. This both collects and concentrates the analyte in the incoming gas stream. The silicon nitride membrane supports a thin metal heating element. After a gas collection phase lasting 30-60 seconds, the preconcentrator membrane is heated up and the analyte is driven out and moved into the micro-GC column. The preconcentrator membrane has a very low heat capacity, and the heating pulse required to drive the analyte out of its sol-gel coating lasts less than one second.

The micro-GC column is a long, small diameter tube next in line downstream from the preconcentrator. The micro-GC is typically around 1m long and is fabricated to occupy about $1cm^2$ of surface area in a silicon wafer. The walls of the GC column are coated, and the selective absorption and desorption as the gas travels through the column cause the various constituent compounds in the gas analyte to emerge from the GC outlet at different times. The pulses of analyte gases that emerge at differing times give the system its chromatographic measuring capability. The outlet of the GC column connects to the coated SAW detector. The preconcentrator, the GC column, and the SAW detector are combined via packaging that handles the microfluidic gas flow as well as the electronic controls and signals (fig. 3). These fit together in a modular fashion for easy assembly and maintenance.

The SAW detector measures the chemicals separated in time by the GC column. It effectively measures the mass of each pulse of analyte as it emerges from the GC outlet and before it is forced out via an exhaust tube. The SAW does this by selectively absorbing and desorbing into its coating the constituent gas components as they pass over the top of the coated portion of the SAW. Extensive optimization work has been done on the preconcentrator, the GC column, and the coatings used on the surface of the SAW. These efforts have been previously reported in [1][2][3].

II. Overview

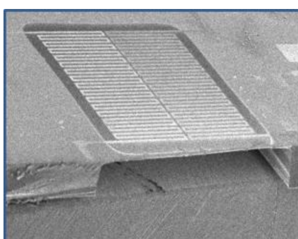
The specific focus of our work, described here, is optimization of the SAW detector and its associated control electronics and mechanical packaging. The Microchemlab chemical sensing system was initially created about 14 years ago and aspects of it were optimized for a number of years after that. We recently reviewed the entire Microchemlab design and concluded that a systematic optimization approach had not originally been done to obtain the best overall performance. We found that a number of design choices had been made based on incorrect assumptions about prior optimization work. In the course of reviewing the design, we decided to examine several key SAW performance criteria to see if substantial improvement in overall chemical sensitivity could be achieved.

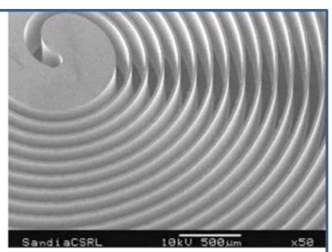
Our optimization efforts included both specific SAW performance criteria as well as ancillary electronic circuitry

* Sandia National Laboratories is a multiprogram laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.

issues. The SAW sensor forms the heart of the detection system. The SAW is an acoustic device that uses electric to acoustic transducers on both its input and output. The acoustic delay through the SAW device is a sensitive function of, among other things, mass loading of the surface of the SAW. The surface of the SAW is coated with a chemically selective absorbing material. Mass changes in the chemically absorbing material register as velocity changes to the acoustic wave travelling underneath it. This can then be detected as either a phase or a frequency difference in the detection electronics.

Figure 1: First Microchemlab unit referred to as the "gold box".





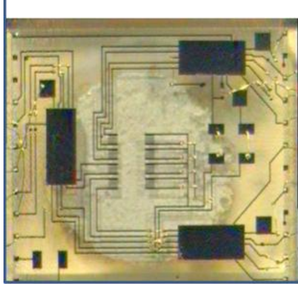


Figure 2: A silicon nitride micro-machined preconcentrator (upper left), a micro-gas chromatograph (GC) column fabricated in silicon using deep reactive ion etching (upper right), and a coated surface acoustic wave (SAW) device mounted in a multi-chip module along with support electronics (lower left).

In order to optimize the performance of the SAW as a detector, one must optimize the SAW device itself as well as its support and detection electronics. The SAW should have low insertion loss and should be a good impedance match to its support electronics. In addition, its output should not drift significantly with temperature variations. The detector support electronics should measure the SAW's phase variation under varying mass loading conditions in the most stable manner possible. In addition, the measurement electronics should introduce the minimum possible noise to the phase shift measurement.

III. THE SAW DEVICE

The central element of our chemical detection system is the SAW detector. The SAW detector consists of a pair of electric-to-acoustic transducers and an acoustic coupling medium. Electrically, the SAW detector appears as a band-pass filter. That is, it has very low insertion loss in a portion of the frequency spectrum (i.e. the passband), and it has very high insertion loss in another portion of the spectrum (i.e. the stopband).

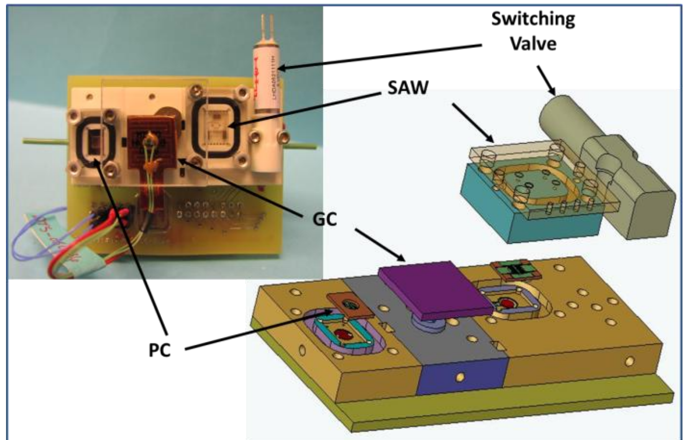


Figure 3: Modular assembly of micro-fluidic, micro-electronic components

The SAW detector is built on a piezoelectric substrate. The electric-to-acoustic transducers are fabricated using two sets of interlaced metal fingers deposited on the surface of the piezoelectric substrate. Each metal finger structure is referred to as an interdigitated transducer (IDT). An alternating voltage applied to the two halves of the IDT will induce a mechanical distortion on the surface of the piezoelectric material. This mechanical distortion will propagate outward away from the IDT as a wave. Proper orientation of the underlying substrate can enable this wave to propagate as a surface-confined Rayleigh wave with little loss or dispersion. Placing a second IDT in the path of this wave creates a two-port electrical device which can be designed to respond electrically with the desired band-pass configuration.

As with any two-port band-pass filter, the SAW filter has parameters of insertion loss, input and output impedance, and stop-band loss. The input and output impedance are determined by the design of each IDT and the coupling coefficient of the IDT metal fingers to the piezoelectric substrate. The electrical input and output impedance of each IDT relate directly to the microwave scattering parameters (S-parameters) S11 and S22, respectively. It is important to design each of the IDT impedances to be 50Ω at the center frequency of the SAW. This provides optimum matching to most microwave test equipment as well as most commercially available components that might be used in a circuit design. We therefore chose 50Ω as the design impedance of our transducers. It is straightforward to design the IDT to attain the desired frequency. The frequency of the IDT is inversely proportional to its finger spacing and is related by the familiar relation $f = v/\lambda$, where v is the acoustic velocity of the substrate material, f is the frequency, and λ is the wavelength.

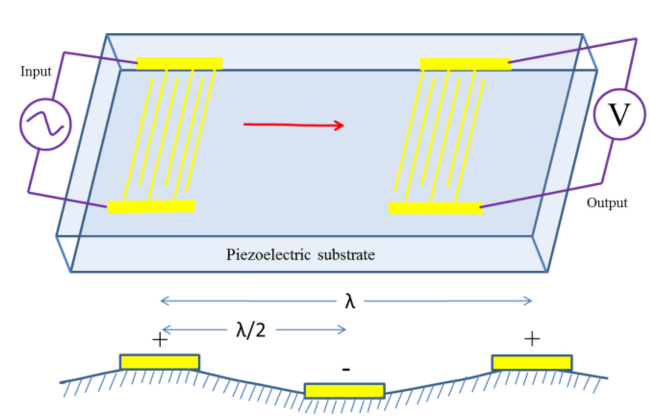


Figure 4: Basic Surface Acoustic Wave Filter

The insertion loss (IL), bandwidth, and stop-band loss of the SAW device are more difficult to design. The nature of the Rayleigh wave in a properly oriented crystal substrate is to be confined to a shallow surface channel of about the width of the transducer. In a 2-port device, this means that the wave emitted from an IDT will propagate equally in two directions, as a forward and a backward component. With this approach, half of the transmitted power will be lost on transmission and another similar proportion will be lost on interception at the output IDT. The insertion loss of such a design can never be better than about -12dB.

To improve the insertion loss of the SAW device, reflectors can be added behind each IDT to create a resonating structure. Then, each successive pass of the wave between the reflectors will tend to couple some energy out. There are two general approaches to creating a resonating acoustic cavity in a SAW device. The first approach is to create an acoustic Fabry-Perot resonator. This is done by adding multiple reflecting fingers made of metal or etched as grooves in the crystal surface. These are placed behind each IDT to reflect the backward travelling wave and resonantly couple it to the forward travelling wave. This design is referred to as a "resonator" approach, since it uses an acoustic reflector placed behind each IDT to create the resonant acoustic cavity.

Each finger of the IDT in the resonator designs that we implemented makes use of a split-finger design. That is, each finger is divided into two λ/8 sections separated by a distance of λ/8. Doing this causes reflections generated from the leading edge of one part of any finger to be phase shifted by 180° by the time it reaches the leading edge of the other half of the split finger. In this way, the reflections from each edge are cancelled out by the reflections from the other half of the split finger.

The second approach to creating a resonant acoustic structure makes each finger pair of the IDT into its own reflector. That is, each finger of the IDT is designed in such a way that it transmits its wave only in one direction. Similarly, any wave that such an IDT intercepts will either be absorbed or reflected backwards. Such a transducer is referred to as a single phase uni-directional transducer (SPUDT). The type of SPUDT that we selected to compare to the resonator design is known as a floating electrode uni-directional transducer (FEUDT) design. The FEUDT has an IDT design in which each finger-pair serves both as a transmitter and as its own resonant reflector. Each finger-pair of the IDT contains a pair of shorted fingers and a pair of open fingers in addition to the usual pair of driver fingers.

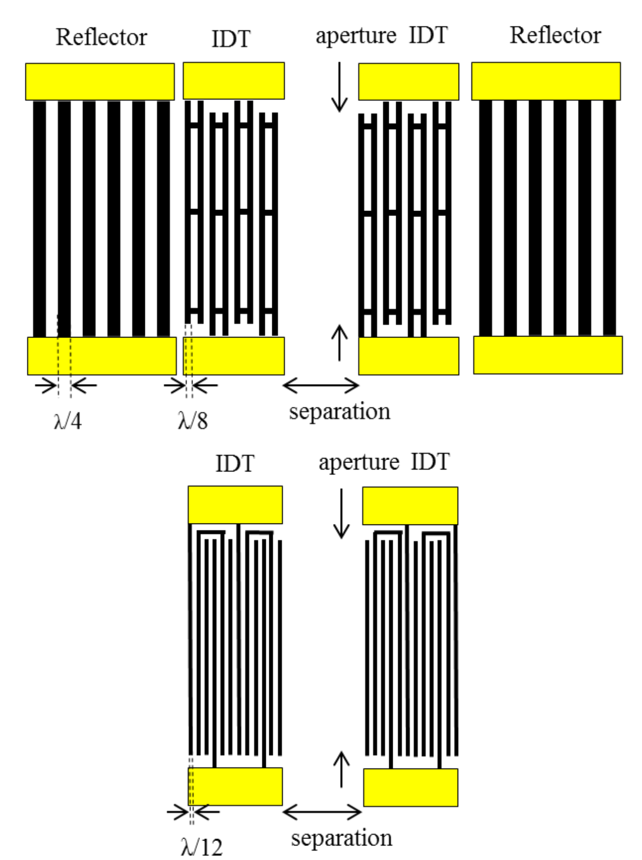


Figure 5: Resonator (top) and FEUDT (bottom) designs.

We created mask sets for fabrication of enough different resonator and FEUDT SAWs to answer the following design questions: (1) Which architecture gives better detection performance, the resonator or the FEUDT? (2) What is the SAW aperture width to give optimal performance? (3) What is the best separation between the IDTs? (4) How much coating area is needed for optimal overall detector performance? (5) What is the best operating frequency for a SAW chemical detector?

IV. CHEMICAL SENSING WITH SAW FILTERS

Confinement of the Rayleigh wave's energy into the thin surface region of the SAW device enables the creation of a sensitive mass detector. To create a measurement system, two SAWs are typically used, a measurement and a reference device. These devices are identical SAWs kept in close physical proximity under identical conditions of temperature and exposure to the gas being measured. Both of these SAWs must be designed in a manner to provide a physical separation region between the SAWs two IDTs. The shape and size of this space between the IDTs largely determines the physical dimensions of the SAW. That is, length of this distance determines the separation between the IDTs, the resulting phase shift, and, to some extent, the insertion loss of the entire SAW. The width of this distance is equal to the width of the IDTs.

A selectively absorbing chemical coating is applied to the space in between the IDTs of the measurement SAW. The application of the chemical coating necessarily unbalances the two SAWs in some degree. The coating should be kept thin to maintain an insertion loss degradation of no more than about 3dB. If the insertion loss difference between the two SAWs was imbalanced by more than this, then an attenuator was used on the output of the reference SAW to bring its insertion loss into line with that of the measurement device.

To determine the optimum SAW design configuration, wafers with 12 different resonator designs and 24 different FEUDT designs were designed and fabricated on Y propagation, Z- cut lithium niobate (fig. 6). The resonators were designed at frequencies of 510 and 960MHz, with IDT separations of 50λ, 100λ, and 150λ, and with IDT aperture widths of 100λ and 200λ. The FEUDT devices were designed at frequencies of 500 and 900MHz, with IDT separations of 100λ and 200λ, with IDTs of 35, 70, and 140 finger-pairs, and with IDT aperture widths of 100λ and 200λ.

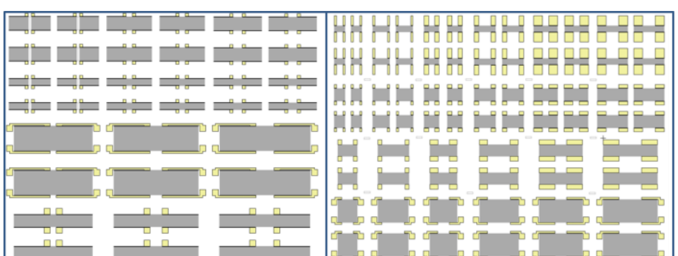


Figure 6: SAW resonator mask set with 12 variants (left) and FEUDT SAW mask set (right) with 24 variants.

The purpose of the variations in frequency is to determine what improvement, if any, one might obtain by increasing the operating frequency of the SAW detector. The purpose of the variation in IDT separation is to determine the effect on magnitude of either phase or frequency shift due to path length under the coating. The purpose of the variation in IDT aperture is to determine the effect on overall coating area and IDT input impedance. The reason for including varying numbers of finger-pairs in an IDT used in the SAW is to attempt to obtain IDTs with input impedance as close as possible to 50Ω. Design equations and models are not reliable for creating IDTs at frequencies above 100MHz, so it is necessary to either work from previous design experience or to create multiple designs based on reasonable estimates of design performance.

V. DETECTION ELECTRONICS

There are two general modes in which the coated SAW device can be operated as a chemical sensor: frequency or phase detection [4]. Both approaches have advantages and disadvantages. Comparison of these two approaches and selection of the optimal approach were important goals in our work. These two different circuit architectures are shown in figure 7.

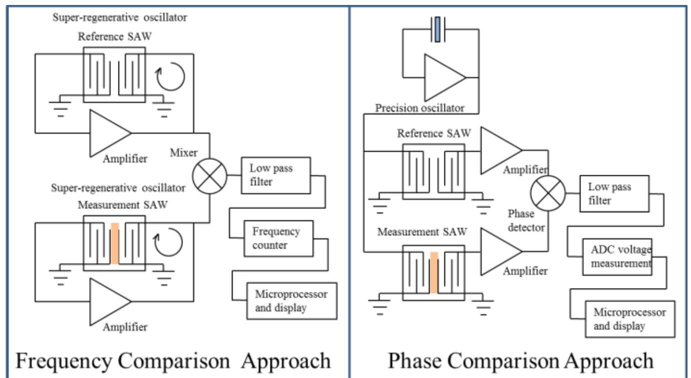


Figure 7: Two Circuit Architectures for SAW-based Mass Sensing

In the frequency comparison approach, shown on the left side of figure 7, the upper SAW forms part of a reference oscillator while the lower SAW is used in a measurement oscillator. The SAW device used is typically a delay-line type two-port microwave device capable of providing feedback only within a narrow frequency band. Ideally this SAW filter should have the bandpass characteristics of a delta function in frequency.

An actual SAW resonator S21 frequency response plot is shown in figure 8. The real SAW has multiple transmission peaks spaced closely together in frequency and with only a small difference in insertion loss. When a SAW with a response similar to that shown in figure 8 is used in a feedback oscillator, the oscillator will have phase noise contributed by the non-resonant peaks. In addition, the oscillator may jump frequency modes as temperature changes or drift cause slight shifts in the frequency dependent loop gain. Another significant disadvantage to the frequency measurement form of chemical detection is that the two oscillators can settle on different modal frequencies. In this case, the offset frequency between the two oscillators may be large while the frequency difference relating to the mass to be measured remains small. This can greatly exacerbate the processing of the frequency output results.

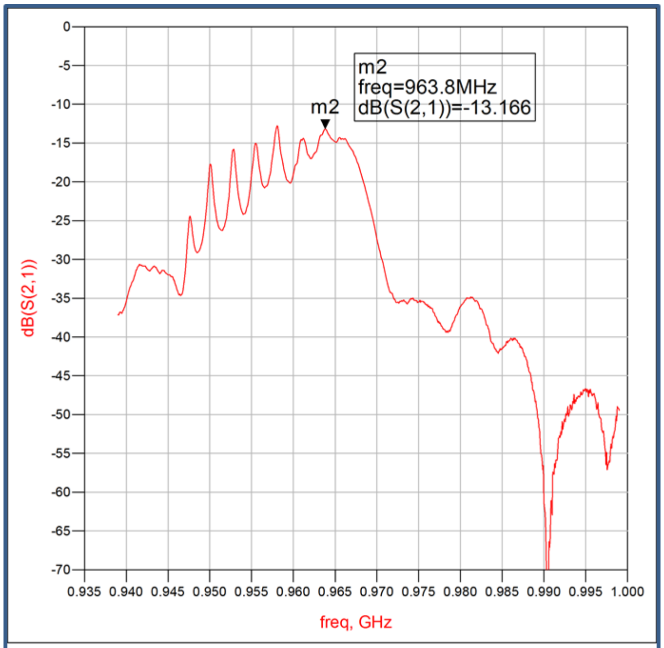


Figure 8: Typical SAW resonator frequency response

There have been two primary advantages posited for using the frequency measurement mode of chemical detection. The first is that it requires less circuitry and fewer components than the phase detection approach. While this may be generally true, it does not provide a good justification if the goal is to produce the most sensitive chemical detection instrument possible. The second perceived advantage is that the frequency measurement approach has an advantage as frequency scales. It has been previously reported in the literature that the change in relative frequency (Δf/f) caused by the addition of mass (Δm) detected by a chemical sensor operated using the frequency detection approach is given by [5]:

$$\Delta f / f = c \, f_o^3 \, \Delta m$$

Where f_o is the unperturbed SAW oscillation frequency, c is a constant of proportionality, and $\Delta f/f_o$ is the magnitude of the relative frequency change. What this implies is that the frequency sensitivity of a chemical detector should improve as the cube of frequency when using a frequency detection methodology. It was stated that “going from 100 to 500MHz will provide more than 100-fold sensitivity enhancement.”[5] when using this method of detection.

We observed a frequency dependence for this form of measurement circuitry given by:

$$\Delta f / f = c \, f_o^1 \, \Delta m$$

With all terms are as described above. This implies that the primary stated advantage for using the frequency measurement approach is incorrect. The disadvantages of using a frequency based form of measurement in SAW-based chemical detectors remain, however.

The frequency shift method remains the most common method of measuring the output of not only SAW-based chemical detectors, but other types as well. Examples of this approach used in a chemical detection system are described in [6] [7] and [8]. However, it is not the best method of measuring the output of a SAW-based chemical detector.

VI. Experiment Design

We made extensive use of two measurement techniques to compare component and sub-system performance. These are S-parameter and metered gas flow measurements. We primarily made use of the microwave network analyzer to collect S-parameter measurements in order to compare SAW device performance. An example of the S-parameters of a SAW resonator is shown in figure 9.

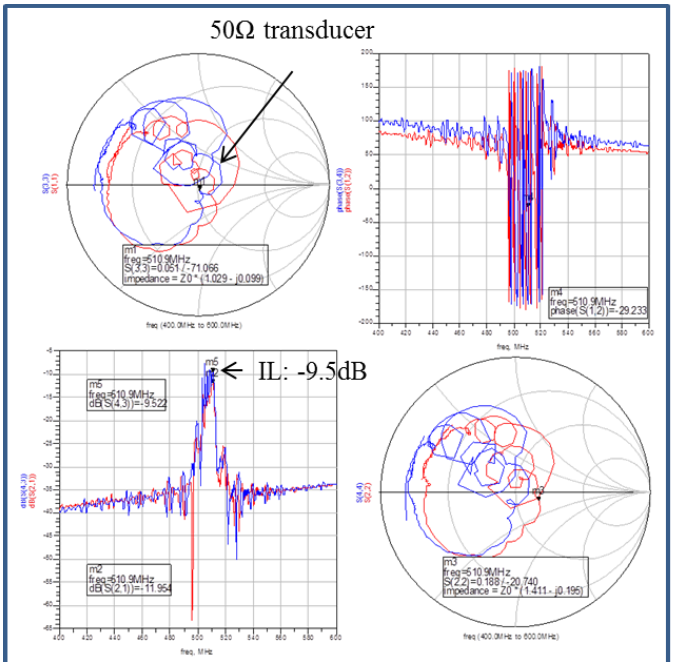


Figure 9: SAW resonator S-parameters, coated and uncoated

S-parameter measurements aid in the evaluation and comparison of different SAW component designs. As was previously stated, our goal was to select the SAW design with the lowest insertion loss and with transducers having impedance as close to 50Ω as possible. The network analyzer directly provides these comparison data via S-parameter measurements.

A metered gas-flow test setup was the measurement configuration that we used as the final arbiter for determining sensor and sub-system performance. The input gas consisted of air with a very dilute analyte, typically at a concentration of about 20ppm. The gas was separated in a GC column and then allowed to flow across the reference and measurement SAW detectors, which were kept in close physical proximity. The outputs of the SAWs were then processed in a variety of different measurement electronics configurations. Our intention throughout the experiments was to separately optimize the detectors and measurement electronics. This was accomplished by varying each of the parameters discussed one at a time.

VII. Conclusions

Armed with our test results, we have been able to greatly improve the performance of our chemical measurement system, Microchemlab. Using a GC column without a preconcentrator, we have obtained detection thresholds of 4ppb from our best SAW detector/measurement electronics combinations. The addition of a preconcentrator can improve this performance by anywhere from 20x to 1000x, depending on the amount of time used to collect the air sample and the degree of impact to the quality of the gas chromatography. Higher levels of preconcentration of the input gas stream can degrade the amount of separation between analyte constituents The changes that we have made to date constitute an improvement factor of over 10,000x in the sensitivity of the best previous performance of the Microchemlab. We believe that further substantial improvement is still possible.

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