AUTORANGING COMPENSATION FOR VARIABLE BASELINE CHEMICAL SENSORS

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ABSTRACT

This paper addresses the need for a broad-base signal conditioning module designed to process chemical sensor signals in such a way that the output of the conditioning circuits ensures similar baseline and dynamic range, regardless of fabrication variation and sensor drift. These baseline compensation circuits are demonstrated in the context of processing resistance changes from composite polymer chemical sensors and tin-oxide chemical sensors. Because of the initial highly variable baseline state of chemiresistors, a large number of bits in an A/D converter are required to translate the sensor information from an array of these sensors into a digital format for use by a microprocessor. In this work, we present a generic circuit for auto-calibrating and compensating for the baseline of a variety of chemiresistive devices in order to improve concentration measurement resolution and analyte discrimination. The measurement circuits optimize sensor resolution via baseline compensation. Dynamic range is standardized to a constant size regardless of initial baseline resistances. The resulting dynamic range can be as much as two orders smaller than an uncompensated circuit and achieve the same sensor accuracy. Simulations have also shown a factor of 68 improvement in resolution.

Keywords: Chemical sensors, Microsensor arrays, Auto-Calibration, Baseline Compensation

1. INTRODUCTION

For chemical vapor monitoring, a clear need exists for a low-power mobile chemical sensing platform capable of detecting a wide variety of chemical analytes and analyte concentrations. Potential applications include food monitoring (by detecting the outgassing components)¹, indoor air quality monitoring, and environmental monitoring. Chemical environmental sensing is particularly important in judging the impact that humans have on the environment. To be effective, chemical sensing must be carried out in a real-time, distributed, and highly sensitive manner. By looking at the local effects of atmospheric pollutants, we can better understand the causes and effects of atmospheric pollution on the regional and global scale.

One approach to monitor chemical environmental impacts is via satellite observation. However, most earth-observing satellites acquire mainly image data. For example, the LANDSAT series of spacecraft acquires detailed images of various places around the world to be compared with earlier images in order to judge human impact and climate change. Images tell us little about the chemical composition of air on the ground level and the environmental impact that the chemical constituents of air can have. Other instruments such as the MOPITT (a gas correlation spectroscopy sensor) instrument on the TERRA satellite are optimized to detect only certain gases (carbon monoxide and methane in this case) and have limited spatial resolution (22km)². Likewise, weather stations are not normally distributed in a very dense manner, depending on location. Also, weather stations are at the present limited in what data they collect (wind speed, temperature at various depths, and barometric pressure). In addition, weather stations are also limited to taking data at fixed points. Although, satellites and weather stations have very useful purposes, a clear need remains for a low-power mobile chemical sensing unit. Previous attempts to build such devices have been limited by a number of factors such as power consumption, the need for a bulky computer to perform processing, and sensor technology issues like variable baseline and drift. A few research efforts have demonstrated techniques for improving portable instrument viability by reducing the inherent variation in chemical sensors with baseline compression techniques. Apsel *et al* uses an adaptive,

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programmable amplifier to stabilize chemiresistors at a predetermined baseline value using floating gate analog memory and novel filtering techniques. The floating gate capability allows a continuous range of programmable values, theoretically enabling perfect baseline compensation (within the constraints of transistor performance); however, these circuits also contribute some distortion to the sensor response as well, generating different response curves for the same analyte, same type of sensor, but different baseline resistances³. Using a different circuit design approach, Neaves and Hatfield have constructed an ASIC specifically designed to extract response signals from an array of polymer chemiresistors. This circuit results in fundamental distortion of the sensor signal by creating a non-linear response of the output current in relation to the input concentration. The ASIC is a versatile generic module for sensor resistance preprocessing, but contains bulky amplifiers and multipliers that may limit usefulness for large arrays in portable instruments in terms of space and power consumption⁴.

Similar to these other research efforts, this work addresses directly the development of a circuit capable of compensating for a variable sensor baseline and sensor drift and suitable for portable chemical sensing instruments. Fundamentally, many chemical sensor technologies, especially chemiresistors, exhibit large changes in baseline resistance. The signal of interest is a small change in resistance overlaying the large variation in initial or baseline resistances. It is desired to extract a differential measurement between the baseline state and the chemically excited state. The initial state of such sensors is not normally known a priori. In order to accommodate the full set of possible initial states (variability in the common mode signal) and the subsequent changes from this state (sensor dynamic range), a wide dynamic range is needed. To convert this analog range into a digital range to be further analyzed by a decision-making model implemented in a microcontroller or similar digital computer, a large number of bits is needed to achieve a reasonable resolution. An analog to digital converter (ADC) with a large number of bits has distinct disadvantages over lower resolution counterparts. For example, a 14-bit ADC can consume up to 20 times more power than a 10-bit ADC of comparable technology. ^{5 6} Higher resolution ADCs also consume more space and are more expensive.

In addition to enabling a lower-resolution ADC in subsequent signal processing, the type of baseline compensator circuit that we present here can compensate for drift. Drift, also referred to as (very) low-frequency noise, is caused in part by the incomplete release of analyte vapor after an experiment has ended. Because the rate of chemical sensor drift in general is much slower than the rate of chemical sensor reaction to a present analyte, the baseline compensator merely needs be reset to the virtual baseline periodically. The problem then becomes one of optimizing the drift reset frequency in the software, which is easily changed to accommodate a wider variety of situations.

2. SENSOR MODELING

In order to maximize the usefulness of a variable-baseline and drift compensation circuit, it should be capable of performing compensation on a wide variety of different chemical sensor types. In this paper, we evaluate our baseline compensation scheme on two types of chemiresistors.

2.1 Composite polymer sensors

A popular chemiresistor is the composite polymer film that consists of an insulating, chemically sensitive polymer and a conductive layer. Composite polymer sensors are organic polymers with implanted conductive carbon-black particles. The polymers in the sensors swell reversibly in response to vapor exposure. Using different organic polymers in different combinations in the manufacture of these sensors causes the sensors to respond to different types of analytes in different ways. When a chemical is applied to which the sensor is sensitive, the polymer swells. In swelling, the deposited carbon-black particles are forced to move farther apart, thereby changing the effective conductivity of the sensor⁷. This change in conductivity can be measured as a resistance and correlated to chemical vapor concentration.

In general, it has been observed that there are two processes, diffusion and percolation, by which a fluid is able to move through a disordered medium. In these sensors, the process of percolation dominates diffusion, due partially to the fact that it is possible for particles to become trapped in the sensor lattice. The process of percolation helps to relate the swelling of the sensor as caused by an absorbing analyte to the change in its conductivity. Initially the resistance of the sensor resembles that of the conductor carbon-black, but as swelling occurs and conduction paths through the carbon-black decrease, the sensor resistance then approaches that of the insulating polymer. A sharp transition point called the percolation threshold occurs when all conduction paths through the carbon-black have disappeared, leading to a sharp transition in resistance⁸. Assuming only two discrete conductances are possible in the sensor, a formula for the volume to resistance relationship may be quantified using a binomial probability distribution as:

$$R = \left(\frac{L}{A}\right) \frac{(z-2)\rho_c \rho_m}{B+C + \left[(B+C)^2 + 2(z-2)\rho_c \rho_m\right]^{1/2}}$$
(1)

$$B = \rho_c [-1 + (z/2)(1 - (v_c/f))]$$
⁽²⁾

$$C = \rho_m[(zv_c/2f) - 1]$$
(3)

where L is the length of the sensor resistor; A is the cross-sectional area of the sensor resistor; z is the number of conductances connected to each node in the lattice, also known as the coordination number; f is the packing factor (ranging from 0.52 for simple cubic spheres to 1 for maximum packing); ρ_c is the resistivity of the carbon-black particles; and ρ_m is the resistivity of the insulating polymer. The input to the equation is v_c , the volume fraction of carbon-black particles. Many other factors have also been known to affect swelling and corresponding resistance including temperature, pressure, and electron tunneling^{8 9}.

The sensor response becomes less and less reversible as it is pushed beyond the percolation threshold. Normally, these sensors are operated at low chemical concentrations where a fairly linear relation between the chemical concentration and sensor resistance exists. In addition to their linear response characteristics, composite polymer sensors are also cheap and easy to manufacture. The small-signal response of this type of sensor may be characterized by ¹⁰:

$$\frac{\Delta R}{R_o} = k[C] \tag{4}$$

where R_0 is the baseline sensor resistance; ΔR is the change in resistance from the baseline; [C] is the applied vapor concentration, typically in ppm; and k is a sensor constant. This relationship demonstrates that the baseline value of resistance does not affect the slope of the response curve (only k affects the slope). k may change with the amount of carbon black in the film or the polymer film thickness, but these factors are not considered here. k does depends on the chemical applied and the type of polymer being used. Typical values of k are 0.00004/PPM for the polymer poly(ethylene oxide) and applied chemical cyclohexane, and a value of 0.0008/PPM for the polymer poly(carbonate bisphenol A) and applied chemical toluene¹⁰.

2.2 Tin-oxide chemical sensors

Tin-Oxide is an n-type semiconducting metal oxide whose conductivity properties are highly sensitive to gases present in the environment. Oxygen species in the thin film that make up the sensor build dangling bonds and other lattice vacancies. Reducing gases in the environment combine with oxygen in the thin film to enable the change in sensor conductivity and resistance¹¹ ¹². Using a necessary heating element, the tin-oxide sensors' resistance can be measured as a response to certain types of gases. The input-output equation may stated as below:

$$\frac{1}{R} = \frac{1}{R_o} + a[C]^r \tag{5}$$

where *a* is a sensitivity coefficient; and *r* is a power law exponent for oxides. A strong dependence on temperature and humidity has further been observed and characterized in^{11 12}.

3. METHODOLOGY

In extracting chemical concentration information from the chemiresistors, it is important to have at hand an efficient and adaptable circuit. The circuit should be capable of interacting with the sensor while adapting to the wide range of sensor characteristics across different methods of fabrication and variable sensing environments. In the case of the tin-oxide sensors and composite polymer sensors, the circuit must be capable of measuring a small change in resistance over a large, but unknown initial resistance. Standard methods using a Wheatstone bridge cannot be applied because the initial resistance is unknown. A Wheatstone bridge requires the bridge to be initially balanced before a change in resistance can be measured. In addition, the sensitivity (the change in the output given a fixed change in the input) is not constant, especially for large deviations from baseline¹³. Although not necessary, a constant sensitivity is often a desirable characteristic of a chemical sensing system. In particular, in a hybrid system, preprocessing modules must transform each type of sensor signal into one that has similar dynamic range and speed limitations for subsequent processing.

A circuit has been fabricated at the University of Washington that is capable of handling the varying parameters described above using discrete components and manual calibration. An integrated circuit adjustable current source (LM334) is used to apply an amount of current necessary to produce some initial output voltage, which is constant between sensors. The current generated by the LM334 is adjusted by manually changing a connected potentiometer. The current then remains constant, but subsequent changes in the sensor properties as caused by an applied chemical will caused the output voltage to deviate from its baseline value. This circuit has been fully tested and shown to work with both polymer and metal-oxide chemical sensors. An integrated version of this circuit is currently in fabrication.

The baseline compensation scheme can be further improved by adding auto-calibration capability. For auto-calibration, a feedback loop is used (Figure 1). This scheme enables a user to simply insert a sensor into the circuit and reset it automatically to compensate for a sensor replacement or sensor drift. The level of current necessary to achieve the initial voltage is automatically applied.

In the baseline compensation scheme, an 8-bit counter is reset and begins to count up in binary. A voltage ramp is produced at the Digital to Analog Converter (DAC) output. The output stage converts this voltage ramp to a current ramp input to the sensor. The current ramp causes the voltage across the sensor to increase (for both sensor types described here). Once the voltage across the sensor reaches a preset threshold value, the comparator shuts off the counter, thereby freezing the amount of current flowing into the sensor. Regardless of the initial sensor state, the initial voltage across the sensor resistor will be predetermined by the threshold, and will fluctuate around this value as chemicals are introduced. After calibration, the counter is disabled to prevent undesired compensation.



All components of this compensation circuit are straight forward except for the output stage. The circuit for the output stage is shown in Figure 2 and Figure 3 demonstrates the entire compensation process. The circuit is a modified instrumentation amplifier acting as a voltage-controlled current source. Op-amp LM324_4 is bootstrapped to require that the voltage across R_x be equal to the applied voltage from the DAC. Ideally no charge flows into the op-amp input so the voltage across R_x will directly produce a current which flows entirely into the sensor, producing the sensor output voltage. Therefore,

$$V_{Sensor} = \frac{R_{Sensor}V_{DAC}}{R_x}$$
(6)

for the chemiresistor. Resistors of size $20k\Omega$ were used because their resistance was large enough to limit power consumption, but not so large that their impedances would begin to produce significant non-idealities in the op-amps by degrading the op-amp input impedance.





Taking the compensation loop into account, the transfer characteristic for the composite polymer sensor changes as shown:

range baseline resistances that could be compensated.

$$Uncompensated \Rightarrow \frac{\Delta R}{R_o} = k[C] \quad and \quad \Delta R = R - R_o \Rightarrow R = R_o + R_o k[C]$$
(7)

$$Compensated \Rightarrow V_o = I_{Set}R = I_{Set}R_o \left(1 + \frac{\Delta R}{R_o}\right) = I_{Set}R_o (1 + k[C]) = V_b (1 + k[C])$$
(8)

The compensated curve produces curves having a constant sensitivity of $V_b k$, unaffected by the baseline resistance. The resulting reduction in common mode variation is demonstrated in Figure 4. Before compensation, the baseline resistance of the poly(ethylene oxide) chemiresistor varies from 25k to 300k. The sensor resistance is extracted as a voltage injecting a constant current into the resistor and measuring the resulting voltage across the resistor. For example, using a constant current of 50 μ A and assuming that the sensor resistance changes by as much as 20% in response to an analyte, the range of output voltages possible is 1.25 V to 18 V. With a 12-bit analog-to-digital converter (ADC), the minimum detectable change in either sensor is 81.8 Ω (0.327% to 0.027%).

In contrast, when using compensation, the minimum detectable change in the $25k\Omega$ and $300k\Omega$ sensors respectively is 1.2Ω to 14.6Ω (0.005%), produced from a voltage range of 2 to 2.4V. In other words, without compensation, an 18-bit converter is required to achieve the same signal resolution as the 12-bit converter used for the baseline-compensated circuit. Compensation provides up to a factor of 68 improvement in response (e.g. concentration) resolution for the same sensors and electronic processing. Likewise, the minimum detectable change in concentration of cyclohexane for the non-

compensated circuit is 16.4 ppm, while for any of the compensated sensors, the minimum detectable concentration change is 0.25 ppm. The compensation circuit has enhanced the performance characteristics of these chemical sensors.



Three Composite Polymer Sensors with different baseline resistances are put into a simple current source circuit in (a) and are compensated in (b). Using the compensation circuit, sensor variability is reduced from 16.75V to 0.4V, thereby reallocating almost all of the dynamic range of the electronics to the sensor response (as opposed to baseline variations).

A similar analysis can be performed on the tin-oxide chemiresistor. The compensated input-output equation reduces to:

$$V_o = V_b \left(\frac{I_{set}}{I_{set} + V_b k[C]^r} \right)$$
(9)

It can be seen here that, at zero concentration of analyte, the output voltage becomes the baseline voltage and that as the analyte concentration increases, the output voltage decreases towards zero.

In a good sensor interface circuit, it is the sensor noise that limits the minimum detectable value and not the circuit into which the sensor is placed. The thermal noise associated with a composite polymer sensor of $100k\Omega$ should be about 0.129 mV in a typical circuit with a bandwidth of 10MHz. With a 12-bit A/D converter, our minimum detectable voltage change is 0.098 mV. Thus the noise limits the minimum detectable value, further adding validity to this design.

4. EXPERIMENTAL RESULTS

Empirical results are collected by placing the compensation circuit and chemical sensors in an enclosed chamber approximately 1 meter in length on each side. The types of sensors used include two types of composite polymer sensors (provided by Nathan Lewis at CalTech) and three types of tin-oxide sensors (purchased from Figaro Eng). A small jar is placed into the test chamber containing the analyte of choice. The jar is separated from the chamber by a solenoid valve. Before any analyte is applied to the chamber, the chamber is purged using compressed air. All sensors are allowed to stabilize prior to introduction of an analyte. The solenoid valves are opened for 30 minutes to allow the analyte to diffuse into the chamber, then closed to allow the sensors to return to their baseline values. Typical results for compensated responses for tin-oxide sensors are shown in Figure 5 and Figure 6, and for composite polymer sensors in Figure 7.

In Table 1, measurements are taken of the initial baseline voltage of composite polymer sensors after they have been compensated. The value for R_{set} used for these measurements was $20k\Omega$. In general, as baseline resistance increases and becomes much greater than R_{set} , the compensation accuracy decreases.

| Baseline Resistance | Compensated Voltage | Threshold Voltage | Percent Difference |
|------------------------|------------------------|----------------------|-----------------------|
| 20kΩ | 2.016 | 2V | 0.8% |
| 50kΩ | 2.012 | 2V | 0.6% |
| 70kΩ | 2.060 | 2V | 3.0% |
| 110kΩ | 2.051 | 2V | 2.55% |
| 200kΩ | 2.153 | 2V | 7.65% |

 Table 1: - Measured vs. Ideal Baseline Compensated Sensor Outputs



Figure 5: Response of Tin-Oxide Sensors to Propanol

As the analyte vapor diffuses in the testing chamber, the sensor voltage beings to decrease. The three types of tin-oxide sensors have been optimized to detect different types of gases and therefor they respond at different rates.



differences.



5. DISCUSSION

Some limitations exist for the current circuit design. For example, the resolution to which the sensor is baseline compensated is limited by number of possible states (number of bits on the counter) and by the range of the current ramp (set by R_x) as shown in Table 1. For an 8-bit counter, 256 possible states are evenly divided within the current ramp range. Increasing the range of the current ramp enables the compensation of a wider range of initial sensor states, but at lower resolution. Baseline compensation is also further diminished by non-idealities in the op-amps used throughout the circuit, especially in the case of the composite polymer sensors which typically have a high initial impedance relative to the other sensor types. Initial output impedances of the polymer sensors can easily be on the order of several hundred k Ω . Another

issue with the op-amp is the finite input bias current. The combined bias current that leaks into the various op-amps at the sensor node should be approximately 1μ A and is variable with temperature. In the case of the composite polymer sensors, the bias current passed through the sensor by the compensation circuit is generally below 100μ A. Because the leakage current is a significant fraction of the compensation current for this sensor type, significant degradation in the predicted circuit response is possible. In addition, the present layout of the circuit requiring the sensor to be connected from various op-amp inputs to ground may also degrade the input impedance of the op-amps to which it is connected, further increasing bias currents and limiting the ability of the voltage followers to correctly follow their input voltages. However, Table 1 indicates that even taking into account the various non-idealities of the op-amps, the circuit still performs compensation to a high degree of accuracy.

As shown in Figure 7, a sensor can be periodically recompensated to account for drift in order to maintain a near constant baseline output. Drift rates noted in our experiments were generally limited to a few percent from the original baseline value. In experiments done elsewhere on composite polymer sensors, drift rates of approximately 16% were observed over a 3 month period. This drift has been shown to not affect sensor sensitivity and when compensated, has a negligible effect on sensor accuracy⁷.

Future work on the compensation circuit includes adding averaging, especially for the case of linearly responding sensors, to process homogenous arrays of sensors for a more robust aggregate output. Recall from equation (8) that for composite polymer sensors of the same type, the constant k will be the same for each analyte for one type of sensor. The differing baseline resistance is not a factor in this equation due to the baseline compensation. Therefore, because of the linear response, independent of baseline resistance, responses of different sensors of the same type can be averaged together for a more robust response. Averaging and other statistical methods can also be used to eliminate defective sensors by comparing an individual sensor's response with the average. Finally, to further reduce noise, the signal can be averaged over time using appropriate filtering techniques.

A future integrated version of this circuit is in process. In integrating, many design simplifications can take place to further optimize the circuit for its intended purpose beyond what is possible with discrete chips. Many of the problems associated with the op-amp non-idealities will disappear as a different method will be used to generate the current ramp not involving op-amps. Current dissipation should also drop tremendously from approximately 100mA per sensor now to milliamps or less. Integrated circuits are also smaller than discrete circuits thereby leading to more portability. In this case, the discrete circuit takes up a volume of approximately 1310 cm³ while the equivalent integrated circuit could be expected to occupy approximately 1 cm³. In addition, an integrated circuit will reduce cost. The discrete version costs approximately \$40 per sensor compensated even when mass-production is taken into account, while an integrated chip could be expected to cost only a few dollars and house compensation capabilities for multiple sensors. Finally, since all sensors chosen for this research effort are silicon-compatible, in the long-term, they can be fully integrated with processing circuits, further reducing power, space, and cost at the system level.

Another simplification beyond merely integrating the compensation circuit is to incorporate floating-gate technology in such a way that overcomes the drawbacks previously presented³. Currently the compensation circuit achieves baseline-compensation using an elaborately large feedback loop composed of counters, digital to analog converters and comparators. The current circuit requires that each sensor have its own complete compensation circuit. However, floating gate technology can reduce the size of the integrated compensation circuit by two orders of magnitude by using floating-gate technology as analog memory¹⁴. By keeping the same compensation scheme as described in previous sections, but adding analog memory, the signal will not be distorted as in previous attempts³. Floating gate technology will also allow most of the compensation circuit will enable the optimization of a large number of these chemical sensors into a small, inexpensive, low-power, mobile chemical sensor unit.

6. RELEVANCE TO CHEMICAL SENSING SYSTEM DESIGN

Many possible applications for a portable low-power optimized chemical sensing unit can easily be formulated. For example, distributed chemical monitoring of atmospheric constituents could potentially have the ability to improve weather forecasting, or to better monitor pollution on a local scale and regional scale. After a chemical leak, it might be useful to monitor the distribution gradient as the leak spreads in order to better contain it. By distributing these sensor arrays in a number of places, they could give a detailed picture of the interaction between the global climate and the atmospheric constituents in a number of widely distributed local areas. In monitoring hydrocarbon products such as carbon dioxide and carbon monoxide, these sensor arrays can provide insight into how specific ecosystems respond to environmental change and subsequently affect and are affected by the global carbon cycle. Because of the distributed, portable nature of these sensors, it is possible to make links between regional and global changes in air quality and climate changes. The distributed nature of the sensors could allow industry to self-monitor or enable government to cheaply and efficiently monitor industrial pollution emissions in real-time.

Another category of applications for the proposed sensor array would be in the area of indoor air quality monitoring. This category applies to office buildings, factories, and perhaps even the International Space Station. Currently indoor air quality systems tend to be expensive and consume large amounts of power¹⁵. While these systems are indeed very accurate, the proposed sensor holds many advantages. Due to the inexpensive low-mass nature of these sensor arrays, a great deal of redundancy is possible. A properly optimized sensor array also has the capability to monitor more atmospheric constituents even before being routed into a computer for further analysis. With these sensors distributed throughout the structure, it would be possible to react to a leak or some other sort of detriment to the air quality in a very rapid and targeted manner. Baseline compensation is essential to these schemes to reduce the need for frequent sensor calibration.

7. CONCLUSION

A discrete circuit capable of compensating for variable baseline and drift has been built and tested. The circuit has been shown to perform compensation for two different types of chemical sensors, composite polymer sensors and tin-oxide sensors. In performing compensation, the sensor dynamic range has been greatly enhanced for an increased capability in both detecting analytes present and in measuring analyte concentration.

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