FY04 Field Evaluations of an In-Situ Chemiresistor Sensor at Edwards Air Force Base, CA

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Abstract

In FY04, an in-situ chemiresistor sensor was further developed and evaluated at Edwards Air Force Base. Initial prototyping and testing began in FY02, and several improvements were identified to improve the stability, accuracy, and sensitivity of the sensor. This report documents the testing and evaluation of three additional features that were tested in FY04: (1) localized heating of the sensor to prevent condensation and improve stability; (2) in-situ baselining to improve the accuracy of the sensor; and (3) preconcentration to improve the apparent sensitivity of the chemiresistor sensor. Results of the tests showed that localized heating of the chemiresistor sensor can stabilize the response for long-term applications. However, drift can occur if the polymers are continually exposed to chemicals. Transient detection methods employed in the in-situ baselining and preconcentration methods were shown to overcome the problem of drift. Both methods yielded TCE concentrations that were comparable to measured concentrations in the groundwater.
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1. Introduction

1.1 Background and Objectives

This report summarizes field tests performed in FY04 at Edwards Air Force Base to further evaluate the performance of an in-situ chemiresistor sensor developed at Sandia National Laboratories. A “Work for Others” contract in the amount of $57K was awarded to Sandia to prepare and deploy chemiresistor sensor systems at an Edwards Air Force Base site contaminated with volatile organic compounds (VOCs). Previous studies had been performed by Sandia National Laboratories in FY02 to perform proof-of-principle and beta testing using the chemiresistor sensor (Ho et al., 2002). The purpose of the FY04 tests was to evaluate three new features and processes that were recommended by Ho et al. (2002):

1. Evaluate the use of localized heating of the chemiresistor sensor to stabilize readings in the presence of high water-vapor concentrations (100% relative humidity)

2. Develop and test a method to baseline (or “rezero”) the chemiresistors by introducing clean humidified air to the sensors in situ

3. Deploy and test chemiresistor sensors integrated with preconcentrators to improve the sensitivity

The remainder of this section provides background material regarding the physics of the chemiresistor sensor and its packaging, along with an overview of the test site. Subsequent sections in this report describe the results of each of the three tests.

1.2 Chemiresistor Sensor and Package

The chemiresistor sensor used in the tests at Edwards Air Force Base is essentially a chemically sensitive resistor comprised of a conductive polymer film on a micro-fabricated circuit. The chemically-sensitive polymer is dissolved in a solvent and mixed with conductive carbon particles. The resulting ink is then deposited and dried onto thin-film platinum traces on a solid substrate (chip). When chemical vapors come into contact with the polymers, the chemicals absorb into the polymers, causing them to swell. The swelling changes the resistance of the electrode, which can be measured and recorded using a data logger or an ohmmeter (see Figure 1). The swelling is reversible if the chemical vapors are removed, but some hysteresis can occur at high concentration exposures. The amount of swelling corresponds to the concentration of the chemical vapor in contact with the chemiresistor, so these devices can be calibrated by exposing the chemiresistors to known concentrations of target analytes.
Two unique features exist regarding the chemiresistor sensor package used in these tests. First, the architecture of the microsensor (Hughes et al., 2000) integrates an array of chemiresistors with a temperature sensor and heating elements. Figure 2 shows two chemiresistors that were used in the FY04 studies at Edwards Air Force Base. The chemiresistor array has been shown to detect a variety of VOCs including aromatic hydrocarbons (e.g., benzene), chlorinated solvents (e.g., trichloroethylene (TCE), carbon tetrachloride), aliphatic hydrocarbons (e.g., hexane, iso-octane), alcohols, and ketones (e.g., acetone). The on-board temperature sensor comprised of a thin-film platinum trace can be used to not only monitor the in-situ temperature, but it can also provide a means for temperature control. A feedback control system between the temperature sensor and on-board heating elements can allow the chemiresistors to be maintained at a fairly constant temperature, which can aid in the processing of data when comparing the responses to calibrated training sets. In addition, the chemiresistors can be maintained at a temperature above the ambient to prevent condensation of water, which may be detrimental to the wires and surfaces of the chemiresistor.

Figure 2. Chemiresistor arrays (chips E23 (left) and E40 (right)) developed at Sandia National Laboratories with four conductive polymer films deposited onto concentric-spiral microfabricated circuits. A temperature sensor (perimeter) and heating element (center) are also integrated on each chip.

A second unique feature is that a robust package has been designed and fabricated to house the chemiresistor array (Ho and Hughes, 2002). This cylindrical package is small (~ 3 cm diameter)
and is constructed of rugged, chemically-resistant material. Early designs have used PEEK (PolyEtherEtherKetone), a semi-crystalline, thermoplastic with excellent resistance to chemicals and fatigue. Newer package designs have been fabricated from stainless steel (Figure 3). The package design is modular and can be easily taken apart (unscrewed like a flashlight) to replace the chemiresistor sensor if desired. Fitted with Viton O-rings, the package is completely waterproof, but gas is allowed to diffuse through a GORE-TEX® membrane that covers a small window to the sensor. Like clothing made of GORE-TEX®, the membrane prevents liquid water from passing through it, but the membrane “breathes,” allowing vapors to diffuse through. Even in water, dissolved VOCs can partition across the membrane into the gas-phase headspace next to the chemiresistors to allow detection of aqueous-phase contaminants. The aqueous concentrations can be determined from the measured gas-phase concentrations using Henry’s Law. Mechanical protection is also provided via a perforated metal plate that covers the chemiresistors. The chemiresistors are situated on a 16-pin dual-in-line package that is connected to a weatherproof cable, which can be of any length because of the DC-resistance measurement. The cable can be connected to a hand-held multimeter for manual single-channel readings, or it can be connected to a multi-channel data logger for long-term, remote operation.

Figure 3. Stainless-steel waterproof package that houses the chemiresistor array. Left: GORE-TEX® membrane covers a small window over the chemiresistors. Right: Disassembled package exposing the 16-pin dual-in-line package and chemiresistor chip.

1.3 Chemiresistor Calibration and Sensitivity

The chemiresistors are calibrated by exposing the chemiresistor arrays to known concentrations of analytes of interest. The change in resistance corresponding to different VOC concentrations is recorded. These calibrations can be conducted under a variety of relative humidity and temperature conditions to provide a suite of training sets that can be used when the chemiresistor is exposed to varying conditions in the field.

The sensitivity of these devices depends on the type of polymer used in the chemiresistor, thickness of the polymer film, the amount of carbon particles added to polymer, separation distance between the electrodes, and the type of analyte. A general observation for the chemiresistors developed at Sandia is that the detection limit is on the order of 1/1000th (or 0.1%)
of the saturated vapor pressure of the analyte being detected. For some VOCs, this detection limit is below the maximum concentration limits set forth by the United States Environmental Protection Agency for air and drinking water (U.S. EPA). For example, m-xylene was reliably detected at 1/100\textsuperscript{th} its saturated vapor pressure, or approximately 100 parts per million (ppm) by volume in the gas phase. According to Henry’s Law, this corresponds to ~2 ppm by mass in the aqueous phase, which is less than the 10 ppm maximum concentration limit imposed by the U.S. EPA. However, for TCE, the chemiresistors can detect gas-phase concentrations as low as 100-1000 ppm, which corresponds to an aqueous TCE concentration of ~1-10 ppm. The U.S. EPA maximum concentration limit for TCE in drinking water is 0.005 ppm, well below the current detection limits. Nevertheless, many applications such as pre-screening and remediation monitoring do not require the capability to provide such low detection limits. In addition, this report describes efforts to develop integrated preconcentrators that can increase the apparent sensitivity of the chemiresistor sensors.

1.4 Background on Edwards Air Force Base and the Field-Test Site

Edwards Air Force Base is located in the Mojave Desert, north of Los Angeles, CA (see Figure 4). Edwards Air Force Base is located at an elevation of approximately 2,300 feet (700 m) above sea level, and the average rainfall is approximately 5 inches per year. Average temperatures in the summer range from a low of 65\textdegree F (18\textdegree C) to a high of 95\textdegree F (35\textdegree C), and average temperatures in the winter range from a low of 35\textdegree F (2\textdegree C) to a high of 60\textdegree F (16\textdegree C).

Edwards AFB serves as a flight-test research center, providing research, development, and testing of military and commercial aerospace systems for the United States. Several locations at Edwards AFB have been contaminated with petroleum products and chlorinated solvents that have seeped into the subsurface. Site 19, which is the location of the tests performed in this study, is located southeast of a complex that was constructed in 1958 to house maintenance equipment and engine test racks. Site 19 has an extensive plume of trichloroethylene (TCE) in the groundwater. The plume is suspected of originating from solvents that were used to clean parts during aircraft engine testing from 1958 to the mid-1970s. Spent solvents were drained into a concrete-lined holding pond, and wastewater that did not evaporate from the holding pond was discharged to a soil catchment basin.
Groundwater at Site 19 occurs in a shallow alluvial aquifer zone (< 50 feet below ground surface) and in a deeper fractured bedrock aquifer zone (> 50 feet below ground surface). The groundwater flow direction is east-southeast, and the depth to groundwater ranges from approximately 28-30 feet below ground surface in the vicinity of the observation well (19-OW02) that was used in this study (see Figure 5). Observation well 19-OW02 is screened from 25 to 45 feet below ground surface across the water table. TCE concentrations in the vicinity of this well were recently measured from a number of wells in February and March, 2004, and averaged 480 µg/L in the shallow screened interval (29-45 feet below ground surface) and 2,900 µg/L in the deep screened interval (48-93 feet below ground surface).
2. Localized Heating of Chemiresistor Sensor

2.1 Principles of Operation

In previous tests at Edwards Air Force Base (Ho et al., 2002), the chemiresistor response was seen to be spurious. Additional laboratory tests showed that a likely cause was the operation of the chemiresistors in 100% relative-humidity environments. Condensation and the presence of liquid water on the chemiresistor may have caused erroneous readings and sharp jumps in measured resistances. Laboratory tests showed that by heating the chemiresistor to temperatures a few degrees Celsius above the ambient temperature, condensation could be prevented and readings were stabilized (Figure 6). The purpose of the field tests was to demonstrate the application of localized heating on the chemiresistors.
2.2 **Calibration and Laboratory Analysis**

Two chemiresitors, E23 and E40, were calibrated for the Edwards Air Force Base field tests (localized heating and in-situ baseline method). A third chemiresistor was calibrated together with a preconcentrator for the integrated chemiresistor/preconcentrator tests and is described in Section 4.2.

The first stage in the calibration of the chemiresistors was to calibrate the resistance temperature detectors (RTDs) on the chemiresistor arrays. This was done by placing the chemiresistors and a thermocouple in an oven. The insulated oven was maintained at approximately 50°C and the temperature was allowed to stabilize. The oven was then turned off while the temperatures were recorded. The thermocouple temperature was plotted as a function of the resistance of the RTD on the chemiresistor. Figure 7 shows the RTD calibration and linear regressions of the chemiresistors E23 and E40.
Next, each of the polymers on the chemiresistor arrays were calibrated to various concentrations of TCE in the presence of 100% humidity. Each chemiresistor array consisted of four polymers (see Figure 2). The voltage applied to each heater on the chemiresistors was adjusted to yield a chip temperature of 30°C. The sensors were then connected to an Agilent 34970A datalogger and placed inside a modified Nalgene bottle filled with deionized water. Calibrations were performed under 100% relative-humidity conditions to simulate field conditions. A schematic of the apparatus used for these experiments is shown in Figure 8.

Table 1. Summary of polymers used in chemiresistors E23 and E40.

<table>
<thead>
<tr>
<th>Chemiresistor Array</th>
<th>Polymer</th>
</tr>
</thead>
<tbody>
<tr>
<td>E23</td>
<td>poly(vinyl tetradecanal) (PVTD1)</td>
</tr>
<tr>
<td></td>
<td>poly(vinyl tetradecanal) (PVTD2)</td>
</tr>
<tr>
<td></td>
<td>poly(ethylene vinyl acetate) (PEVA1)</td>
</tr>
<tr>
<td></td>
<td>poly(ethylene vinyl acetate) (PEVA2)</td>
</tr>
<tr>
<td>E40</td>
<td>poly(epichlorohydrin) (PECH)</td>
</tr>
<tr>
<td></td>
<td>poly(N-vinyl pyrrolidone) (PNVP)</td>
</tr>
<tr>
<td></td>
<td>poly(isobutylene) (PIB)</td>
</tr>
<tr>
<td></td>
<td>poly-ethylene vinyl acetate (PEVA)</td>
</tr>
</tbody>
</table>
The chemiresistor sensors were allowed to stabilize in the 100% humidity environment and then various concentrations of TCE were added to the system. TCE vapor concentrations of 250, 500, 750, and 1000 ppm \(^1\) TCE were bubbled through the deionized water and the sensors were allowed to stabilize during each exposure. The relative change in resistance of the sensors during an exposure was found by first taking an average of stable baseline values, \(R_b\), for two minutes prior to the exposure to TCE. Next the value of the resistance during an exposure to TCE was calculated by taking a two-minute average of the resistance values, \(R\), after the response had stabilized. Then the relative change was calculated using Equation (1).

\[
\frac{\Delta R}{R_b} = \frac{R - R_b}{R_b}
\]  

(1)

Univariate and multivariate models were calculated from the resulting relative changes. Univariate models were generated by plotting the relative change in resistance against TCE concentrations (Figure 9). Table 2 shows the resulting univariate regressions.

---

\(^1\) The vapor concentration of TCE is approximately 100 times greater than the equilibrium aqueous concentration (e.g., 100 ppm of TCE in the vapor phase would correspond to an equilibrium aqueous concentration of ~1 ppm.)
Figure 9. Univariate calibration of chemiresistor E23.

Table 2. Univariate calibrations for chemiresistors E23 and E40.

<table>
<thead>
<tr>
<th>Polymer</th>
<th>Regression Type</th>
<th>Regression (ppm)</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>PVTD1</td>
<td>Power</td>
<td>$y = 4.90E+05x^{3.98E-01}$</td>
<td>.990</td>
</tr>
<tr>
<td>PVTD2</td>
<td>Power</td>
<td>$y = 4.61E+05x^{1.02E+00}$</td>
<td>.981</td>
</tr>
<tr>
<td>PEVA1</td>
<td>Power</td>
<td>$y = 2.68E+05x^{2.61E-01}$</td>
<td>.991</td>
</tr>
<tr>
<td>PEVA2</td>
<td>Power</td>
<td>$y = 4.47E+05x^{1.03E+00}$</td>
<td>.986</td>
</tr>
<tr>
<td>PIB</td>
<td>Power</td>
<td>$y = 6.29E+06x^{1.05E+00}$</td>
<td>.649</td>
</tr>
<tr>
<td>PNVP</td>
<td>Linear</td>
<td>$y = -2.07E+05x^{-0.690}$</td>
<td>-.690</td>
</tr>
<tr>
<td>PEVA</td>
<td>Power</td>
<td>$y = 5.36E+04x^{9.18E-01}$</td>
<td>.999</td>
</tr>
</tbody>
</table>

$y = $ TCE vapor concentration (ppm)

$x = \Delta R/R_b$

Multivariate were also generated by evaluating the combined response of different chemiresistor polymers and the temperature to different concentrations of TCE. The multivariate calibrations curves were generated using Statistica™. An example of a multivariate calibration equation is shown below for E23. Because only one analyte was investigated (TCE), the univariate regressions suffice. However, when multiple analytes are present, or when interferences (such as fluctuating temperatures and relative humidity) exist, multivariate regressions can provide more accurate predictions by taking multiple factors and combinations of factors into account.

\[
TCE(\text{ppm}) = -1.61E+00 + 3.71E+07\Delta R/R_{\text{PVTD1}} + 4.56E+05\Delta R/R_{\text{PEVA2}} -2.50+E07\Delta R/R_{\text{PVTD2}} \\
*\Delta R/R_{\text{PVTD1}} - 1.23E+06\Delta R/R_{\text{PVTD1}} * \text{RTD } ^\circ \text{C} - 6.69E+03\Delta R/R_{\text{PVTD1}} * \text{RTD } ^\circ \text{C}
\]
2.3 Field Deployment

Two chemiresistor sensors E23 and E40 were deployed at Edwards Air Force Base’s Site 19 on December 15-16, 2003. This initial deployment was intended to test the localized heating and in-situ baseline method.

Three submersible thermocouples and a KPSI PT700 submersible pressure transducer were also deployed. Two thermocouples were placed inside the bubblers for the in-situ baselining tests and the third was deployed in the groundwater. A program was written (see Appendix A) that monitors all the sensors deployed and incorporates multivariate models of TCE concentrations for each of the sensors. The program also performs diagnostics to ensure that environmental conditions are acceptable for the test procedures. The program checks to ensure that it is between 9 AM and 3 PM before running the pump to ensure that the batteries can be efficiently recharged by the solar panel during daylight hours. It also checks the temperature of the water bubblers to ensure that the relative humidity reaching the sensors will be close to 100% at the groundwater temperature. The program monitors the response of the chemiresistors and logs data once an hour. Since the number of sensor measurements outnumbered the amount of available channels on the CR10X, an AM16/32 multiplexer was used. The AM16/32 multiplexer adds up to 32 differential measurements to the CR10X.

On December 15, 2003, each sensor was wired to the CR10X and the AM16/32 multiplexer in the Earth Tech trailer. On 12/16/03 the two enclosures, sensors, solar panel, deep cycle marine battery and a tripod was moved to Site 19 (Figure 10). Initially, SU44-N04S was the well selected for deployment of the sensors. However, the 2” diameter of the well was too small for the chemiresistor and associated instrumentation to fit in. The entire system was then moved to well 19-OW02, approximately 70 feet to the southwest (see Figure 5). The diameter of this well was 4” and was able to accept all of the sensors with ease. The total depth of 19-OW02 is 47 feet, and the water table in this well was located 31.46 feet below the top of the casing.

![Figure 10. Left: setting up the solar-powered data-logging station at Site 19. Right: data-logging instrumentation inside enclosure.](image)
The submersible pressure transducer was deployed approximately 6 feet into the water. The two chemiresistors were placed down the well just above the water table. Prior to leaving site 19 on December 16, a voltage was applied to the heaters of the chemiresistor sensors to maintain the sensor temperature at 30°C.

A phone line was installed in the nearby trailer and connected to the COM210 modem inside the enclosure. Communication between the data logger and a remote computer (at Sandia) were successfully established. Data was collected for the localized heating tests for about a month, but “external” issues associated with the communication developed thereafter. First, the phone line was disconnected by Edwards Air Force Base for security reasons. It took several months for the phone line to be approved. Then, animals had chewed through the phone line, and the line had to be spliced and inserted into flexible metal tubing to prevent further disruption from animals.

2.4 Results and Discussion

The E40 chemiresistor resistances during a six-week period in February and March of 2005 are shown in Figure 11. The resistances of each polymer (PECH, PNVP, PIB, and PEVA) are plotted as a function of time. Voltage was applied to the on-chip heater to maintain a temperature of approximately 30 °C, and Figure 11 shows the temperature of the chemiresistors during this six-week period as well. The temperature fluctuated by about one degree Celsius due to the fluctuations in applied voltage. During the day, when the solar panel was recharging, the applied voltage would increase slightly. During the night, the applied voltage would decrease slightly.

Figure 11 shows that the measured resistances during this time are quite stable. Previous results (Ho et al., 2002) showed that readings could be spurious when heating was not applied to prevent condensation. The current results confirm that the application of localized heat can improve the stability of the sensors when exposed to 100% relative humidity. The PEVA polymer shows a gradual increase with time. We have seen evidence in the laboratory that under continuous exposure to TCE (or water), the PEVA polymer tends to “creep” upward (Wang et al., 2004). As a result, occasional “baselining” or “rezeroing” may be necessary for continuous operation (see Section 3). In addition, short-duration transient readings may be better suited for in-situ measurements (see Section 4).

Electrical resistances are reported in Figure 11 as opposed to actual TCE concentrations because baseline values are needed to convert the resistances to TCE concentrations (see Section 2.2). The baseline resistance values represent ambient conditions with no contaminants at 100% relative humidity. Because the sensors were deployed directly into the contaminated well from the surface where the relative humidity was less than 100%, no baseline was obtained. An in-situ baselining procedure described in the next section (Section 3) is necessary to provide the baseline resistances that can be used to convert the resistances to actual TCE concentrations.
3. In-Situ Baselining

3.1 Principles of Operation

Long-term continuous exposure to volatile organic compounds (or even high concentrations of water vapor) can cause “drift” in the chemiresistor sensors. Some polymers, such as PEVA, have been seen to gradually increase in resistance, especially when exposed to analytes that have an affinity to the polymer. As a result, an automated in-situ baselining procedure was developed to “rezero” the sensors on a periodic basis. In addition, an in-situ baselining procedure is necessary to convert raw resistances measured by the chemiresistors into actual contaminant concentrations using regressions.

The general principle is to pass clean humidified air over the face of the sensor package, which is presumed to be already located in a 100% relative-humidity environment (e.g., in a well above the water table). Clean air is pumped through water bubblers to saturate the air with water vapor. The humidified air is then passed through Teflon tubes tethered to the sensor cables to the face of the sensor. If no chemicals are present, the introduction of clean 100% relative-humidity air should not change the response of the sensors. However, if the sensor is exposed to chemicals in its humidified environment, the introduction of clean humidified air should reduce the resistance to its original baseline value (with no chemicals present). After the resistances stabilize, the pumping is stopped and the resistances should increase again to a value corresponding to the concentration of the chemical(s) that is present. The change in resistance is used with the calibration curves to estimate the chemical concentration near the chemiresistor.
3.2 **Calibration and Laboratory Analysis**

An in-situ baselining apparatus was assembled by using a 1-1/8-inch inner diameter coolant hose, this diameter provided a snug fit around the chemiresistor housing. A 2-1/2-inch length of tubing was cut and a hole was drilled to accept a Swagelok® elbow adapter. A schematic of the apparatus is shown in Figure 12.

![Figure 12. Left: schematic of fittings for the in-situ baseline apparatus. Right: actual system being deployed down a well.](image)

Clean humidified air was supplied to the face of the chemiresistor using a DC-powered pump and a bubbler filled with clean deionized water. Penn-Plax Silent Air B11 pumps were used to supply the air for in-situ baselining. The air pumps were powered with 1.5 volts supplied from a commercial voltage regulator. The bubbler was constructed out of a glass bottle filled with deionized water and sealed with a lid. Holes were drilled on the lid to accept tubing from pumps. Aerators were used inside the water bottles to aid in saturating the air with water vapor. Figure 13 shows the pump and the bubbler.
Figure 13. DC-powered pumps and water bubblers.

When the pumps are turned on, ambient air is blown through the bubblers and then to the sensor. The coolant tubing ensures that the clean humidified air covers the face of the sensor. A proof of principle experiment was performed to test the effectiveness of this procedure.

The chemiresistor E23 was placed in clean deionized water and the pumps were turned on. Then 5 mL of TCE were added to the 500 mL of deionized water (enough to saturated the water) with the pumps still running. The pumps were then shut off to observe the response of the chemiresistor. After a few minutes large resistance increases were observed, indicating the detection of the TCE. After the pumps were turned back on, the resistances began to drop again as a result of the clean humidified air passing over the chemiresistor face. The results from the experiment are shown below in Figure 14.
3.3 **Field Deployment**

On December 16, 2003, the in-situ baselining apparatus was deployed at Site 19 at Edwards Air Force Base. Two pumps and two water-bubbler jars were placed in an enclosure, which was situated on the tripod holding the data-logging enclosure. The glass jars were filled with clean drinking water and strapped securely in the enclosure (Figure 15).

A 50-foot length of Teflon tubing was attached to each Swagelok elbow on the chemiresistor sensor and connected to the water bubblers, which were connected to the pumps. The sensors
were then lowered into the well. Wires were connected between the data logger and the PennPlax pumps so that the pumps could be remotely operated. Each pump was wired in parallel to one NTE956 variable voltage regulator set to 1.6 V, and power was supplied by an external battery (Everstart Marine DC24-6 75 amp-hour), which also supplied power to the other data-logging equipment.

A program that allowed a user to turn on the pumps remotely was uploaded to CR10X. Once initiated, the program turned on the pumps for 30 minutes and established a baseline value of chemiresistor resistances. The program allows the pumps to turn on only when the saturated vapor pressure of the water bubblers (as determined by the water-bubbler temperature recorded by thermocouples in the bubblers and thermodynamic relations pre-programmed in the data logger) is at least 10% greater than the saturated vapor pressure corresponding to the groundwater temperature (~20°C). This ensures that the air humidified by the water bubblers contains an amount of water vapor that is at least equal to 100% relative-humidity air at the groundwater temperature. Any excess water vapor will condense as the air cools while being passed through the tube to the chemiresistor sensors.

During initial testing at the site, the voltage regulator for the pumps became too hot when the pumps were turned on, and the pumps were automatically turned off. An NTE403 heat sink was added to the voltage regulator, and the pumps were able to run successfully without overheating the voltage regulator.

![Figure 16. Voltage regulator assembly for the pumps with (left) and without (right) a heat sink.](image)

During the second trip out to Edwards Air Force Base (May 10-11, 2004), we discovered a faulty connection between the negative side of the battery and the ground of the NTE956/NTE403 package that was causing a squealing noise from the pumps. Originally the input voltage wire and the ground wire from the NTE956/NTE403 package were simply wrapped around the battery wires and secured with electrical tape. The wires were soldered directly to the battery and the problem was resolved. Additional issues with the program that controlled the in-situ baselining procedure were also corrected during the second trip to Edwards, and the in-situ baselining apparatus was now functioning properly.
3.4 Results and Discussion

The sensors were allowed to stabilize for a week after the second trip out to Edwards Air Force Base. On May 17, 2004, an in-situ baseline procedure was performed from Sandia by dialing into the CR10X and toggling the flag that started the baseline procedure. Figure 17 shows the response of two polymers on the chemiresistor E40 to the baseline procedure.

![Figure 17. Response of polymers PIB and PEVA on chemiresistor E40 to the in-situ baselining procedure.](image)

The polymers PIB and PEVA both decreased during the addition of clean humidified air and returned to a value similar to the pre-baseline values after the pumps were turned off. The response of two polymers on chemiresistor E23 are shown in Figure 18.
Polymer PVTD1 decreased upon the addition of clean humidified air, but PVTD2 increased upon the addition of the clean humidified air. Because we know the water in the well is contaminated with TCE, the addition of clean humidified air should decrease the measured resistances. The reason for the increase in PVTD2 is unclear.

Another in-situ baselining procedure was performed on June 7, 2004, via remote connection to the data logger. The observed results were similar to the first run. Figure 19 shows the response of polymers PIB and PEVA on chemiresistor E40 during the baselining procedure. The polymer PEVA on E40 decreased when the procedure was followed but the polymer PIB on E40 did not show a noticeable response. Both polymers returned to or stayed at current resistance values after the pumps were turned off.

Figure 20 shows the response of polymers PVTD1 and PVTD2 on chemiresistor E23 to the baselining procedure. Both polymers increased upon the addition of clean humidified air and continued to increase after the pumps were turned off. Again, the reason for this increase during the introduction of clean humidified air is unknown. As a result, only the results of E40 were used in the analysis.
The TCE concentration in the vicinity of the chemiresistors was calculated using the relative change in measured resistances during the baselining procedure. Multivariate regressions determined from laboratory testing were used in the analysis. Table 3 shows the predicted TCE vapor concentrations in well 19-OW02 at Site 19 based on the in-situ baselining procedure using
chemiresistor E40. The measured resistances used to calculate the estimated TCE concentrations were taken between 5 and 12 hours after the baselining procedure to allow the sensors time to stabilize. The estimated vapor-phase concentrations are then used to estimate the equilibrium aqueous concentration in the groundwater. Henry’s Law is used to determine the aqueous concentration from the vapor concentration assuming that the liquid and gas phases are in equilibrium. We assume that the Henry’s Law constant for TCE at 20°C is 0.007 m³-atm/mol (Gosset, 1987) and that the atmospheric pressure at Site 19 is approximately 0.2 atm (elevation of 2,300 feet above sea level). The actual measured groundwater concentrations are also shown in Table 3 for comparison.

Table 3. Estimated TCE concentrations based on in-situ baselining procedure.

<table>
<thead>
<tr>
<th>Method</th>
<th>TCE Vapor Concentration (ppmv)</th>
<th>TCE Aqueous Concentration (µg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>May 17, 2004</td>
<td>June 7, 2004</td>
</tr>
<tr>
<td>Chemiresistor E40 in 19-OW02 (PEVA and PIB)</td>
<td>125</td>
<td>128</td>
</tr>
<tr>
<td>Groundwater Sampling**</td>
<td>8 – 85</td>
<td>150 – 1,600</td>
</tr>
</tbody>
</table>

*Aqueous concentrations estimated using Henry’s Law.
**Aqueous concentrations measured in the shallow screened interval from 13 wells at Site 19 (Earth Tech, 2004). Equilibrium vapor concentrations estimated using Henry’s Law.

The TCE concentrations estimated from the in-situ baselining procedure are roughly 40% higher than the highest measured groundwater concentration at the site in the shallow region. A potential reason for the discrepancy includes incomplete humidification of the air used to purge the sensors. If the relative humidity of the air passing over the sensors was less than 100%, the resistances would decrease below the baseline used to calibrate the sensors. The resulting relative change in resistance would then be greater than the corresponding change for a given concentration, and the estimated concentration would be too high.

Despite the discrepancy, it was encouraging to see that the estimated concentrations from the two different runs were quite consistent. As long as the readings are consistent, the absolute discrepancy of 40% can be acceptable for the purposes of monitoring long-term trends and changes in groundwater concentrations.

## 4. Preconcentration

### 4.1 Principles of Operation

The detection limit of chemiresistor sensors for a given analyte is typically 0.1% of the saturated vapor pressure of the analyte. Below this concentration, the chemiresistor may not respond to
the presence of the chemical. In these low-concentration environments, preconcentrators may increase the apparent sensitivity of the chemiresistor.

Preconcentrators that have been developed at Sandia consist of a microhotplate that is coated with a special sorbent. The sorbent absorbs VOCs, even in very low concentrations. After a prescribed loading period, the microhotplate is heated rapidly to desorb the accumulated chemicals. The microhotplate preconcentrator is placed face-to-face against the chemiresistor array so that the concentrated plume of VOC vapor is then detected by the adjacent chemiresistor. The plume is allowed to diffuse out of the package through slots adjacent to the preconcentrator.

![Microhotplate preconcentrator](image)

**Figure 21.** Microhotplate preconcentrator (several millimeters in size). A drop of black sorbent is placed on top of a thin (1 μm thick) silicon-nitride film that can be rapidly heated to 300 °C.

### 4.2 Calibration and Laboratory Analysis

Three chemiresistor/preconcentrator (Chemi/PC) packages were tested prior to the field deployment at Edwards Air Force Base: 1)E57-PC32, 2)E58-PC40, 3)E61-PC41. The fabrication and assembly of the Chemi/PC packages are described in detail in Ho et al. (2003).

The chemiresistor chips where developed using up to four separate polymer carbon depositions. For the purpose of the Edward’s deployment two different polymers were used: PEVA and PVTD with 20% carbon black to 80% polymer by mass. The 20/80 mixture was used to generate a low resistance since this has been shown to respond better to low levels of VOCs (Wang et al., 2004).

The preconcentrator was fabricated utilizing a KOH-etching procedure with a silicon nitride coating. A binding agent (.00764g PIB to 1ml of TCE for a 5% solution) was then deposited on the preconcentrator, (to view a video (4.2 MB) of the deposition process see: [www.sandia.gov/sensor/PC_deposition_7-1-03.mpg](http://www.sandia.gov/sensor/PC_deposition_7-1-03.mpg)). Carboxen 1003, (~325 mesh), was then placed on the preconcentrator and baked for 1-2 hours binding the Carboxen 1003 to the binding
agent. Wires were then connected to the preconcentrator and placed in a custom platform. The housing was epoxied to the chemiresistor. Then the resulting preconcentrator manifold assembly integrated with a chemiresistor was placed in a chemiresistor housing for testing (Figure 22).

![Microhotplate Preconcentrator](image1)

Figure 22. Top: microhotplate preconcentrator held in custom-designed platform. Bottom: preconcentrator integrated with chemiresistor sensor package.

The three packages were then tested under dry and 100% relative-humidity conditions at various concentrations of VOCs. Different concentrations can be accomplished by mixing dry or 100% relative-humidity air with a given concentration of VOC. To achieve 100% relative-humidity conditions, two bubblers were placed before the 6-inch steel-tube apparatuses. Figure 23 shows a schematic of the test apparatus.
A series of 10 5-volt pulses were applied to the preconcentrator to purge the sorbent of any existing VOC. Then the preconcentrator was exposed for 15 minutes and pulsed one more time to produce a plume of VOC to the chemiresistor (Figure 24).

The difference in measured resistances between the baseline pulses and the concentration pulse was used for calibration. Chemi/PC package E57-PC32 was selected based on this testing because it produced the most reliable and consistent results. Three concentrations were used (0ppm, 25ppm, 50ppm) with TCE, which was the VOC of interest in this case. (Note: other tests were conducted with M-Xylene as well).
Figure 25 shows that the PVTD2 polymer on PC32-E57 yielded a distinct difference between 0, 25, and 50 ppm TCE. This was not as obvious with the other polymers in some of the other Chemi/PC packages. When PEVA1 and PEVA2 were tested their results were sporadic and showed no separation between the varied concentrations.

Figure 25. Response of polymer PVTD2 from PC32-E57 for three pulses at three different concentrations of TCE at 100%RH.

A calibration was performed and a linear regression was applied with a corresponding R² value. PEVA1 and PEVA2 produced R² values <.1, while both the PVTD1 and PVTD2 values showed R² values >.85, with PVTD1 generating an R²=.9544 and PVTD2 generating an R²=.8566. Figure 26 shows the calibration graph for PVTD2 PC32-E57 as the pertaining peaks are a function of DeltaR/Rb. The DeltaR/Rb values can be taken from the field and a concentration value can be obtained.
Figure 26. Calibration curve for Chemi/PC package PC32-E57 using peak measured resistance.

4.3 Field Deployment

Sandia visited Edwards Air Force Base on May 10-11, 2004, to deploy the integrated chemiresistor/preconcentrator sensor. The integrated Chemi/PC sensor PC32-E57 was deployed in well 19-OW02, 28 feet below the top of the casing. The measure groundwater depth on May 10 was 31.92 feet below the top of the casing. Testing was performed to ensure that the communications system, pumps (for in-situ baseline testing), voltage regulators (for localized heating of the chemiresistors), and preconcentrators were functioning (Figure 27). The preconcentrators were then tested several weeks later via remote connection from Sandia in Albuquerque. In-situ baseline testing was also performed remotely during the spring/summer of 2004.
4.4 Results and Discussion

Three separate tests were conducted to test the integrated Chemi/PC sensor on July 9, 13, and 15, 2004. A program was uploaded remotely to the CR10X data logger (see Appendix B) that follows the preconcentrator firing procedure shown in Figure 24. The user remotely initiated the process to utilize the preconcentrator and obtain concentration estimates based on the regressions obtained in the laboratory.

During the three tests, the polymer PVTD produced repeatable results. Figure 28 and Figure 29 show the response of PVTD2 and PVTD1 during the desorption (firing) of the preconcentrator for each of the three tests as a function of time. The response of the polymer PEVA was not as distinct as the PVTD polymer, but a response was evident in all three runs (Figure 30). The TCE vapor concentration is estimated from the maximum relative change in resistance measured after the preconcentrator was fired and the analyte was desorbed.
Figure 28. Response of polymer PVTD2 (PC32-E57) due to preconcentrator desorption during three separate runs.

Figure 29. Response of polymer PVTD1 (PC32-E57) due to preconcentrator desorption during three separate runs.
Figure 30. Response of polymer PEVA2 (PC32-E57) due to preconcentrator desorption from three separate runs.

The estimated TCE vapor concentrations are also plotted in Figure 31 for all four polymers. The estimated TCE vapor concentrations from the PEVA polymers ranged from 2 to 15 ppmv. The estimated TCE vapor concentrations from the PVT polymers ranged from 32 to 37 ppmv. The results for each polymer were also quite consistent over the three runs.
The estimated TCE concentrations are tabulated in Table 4 along with the estimated equilibrium aqueous concentrations (using Henry’s Law). The measured aqueous concentrations from groundwater samples taken in February/March 2004 are also shown for comparison. Table 4 shows that the estimated TCE concentrations using the Chemi/PC sensor are within the range of measured TCE concentrations from groundwater samples.

Table 4. Estimated TCE vapor and aqueous concentrations from the integrated Chemi/PC sensor (PC32-E57) along with measured groundwater concentrations.

<table>
<thead>
<tr>
<th>Method</th>
<th>TCE Vapor Concentration (ppmv)</th>
<th>TCE Aqueous Concentration (µg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemiresistor E40 in 19-OW02 (PEVA and PIB)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>PVTĐ</td>
<td>32 – 37</td>
<td></td>
</tr>
<tr>
<td>PEVA*</td>
<td></td>
<td>34 – 260</td>
</tr>
<tr>
<td>PVTĐ*</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Groundwater Sampling** (February-March, 2004)</td>
<td></td>
<td>8 – 85</td>
</tr>
</tbody>
</table>

*Aqueous concentrations estimated using Henry’s Law.

**Aqueous concentrations measured in the shallow screened interval from 13 wells at Site 19 (Earth Tech, 2004). Equilibrium vapor concentrations estimated using Henry’s Law.
5. **Summary and Recommendations**

Tests were performed in FY04 to evaluate three new features and processes associated with chemiresistor sensors that were recommended by Ho et al. (2002):

1. Evaluate the use of localized heating of the chemiresistor sensor to stabilize readings in the presence of high water-vapor concentrations (100% relative humidity)

2. Develop and test a method to baseline (or “rezero”) the chemiresistors by introducing clean humidified air to the sensors in situ

3. Deploy and test chemiresistor sensors integrated with preconcentrators to improve the sensitivity

### 5.1 Localized Heating

Results from the localized heating tests showed that the response of the chemiresistors were stabilized as a result of heating the chemiresistors to temperature above the ambient. This prevented condensation and provided more stable measurements than those observed in Ho et al. (2002). However, some drift was observed in the response of the PEVA polymer as a result of continuous exposure to TCE. Therefore, we recommend that transient responses are needed to accurately characterize concentrations. Two methods that employ transient processes, in-situ baselining and preconcentration, were evaluated.

### 5.2 In-Situ Baselining

An apparatus and method were successfully developed and demonstrated that allowed the chemiresistor sensor to be “re-zeroed” remotely and in situ. Clean humidified air was passed along the face of the chemiresistor to rezero or baseline the sensor. The subsequent change in measured resistance was then used to estimate the concentration. The PIB and PEVA polymers performed best in this scenario, while the PVTD polymers did not respond well to the in-situ baselining procedure. Estimates of the TCE concentration using this method were about 40% higher than measured groundwater samples.

### 5.3 Preconcentration

Preconcentrators were integrated with the chemiresistor sensors to yield a Chemi/PC package that attempted to increase the sensitivity of the chemiresistor by desorbing accumulated amounts of analyte from the sorbent on the PC. Three separate tests showed that the integrated Chemi/PC package functioned successfully and that the measured TCE concentrations were within the range of measured concentrations from groundwater samples at the site.
5.4 Recommendations

Based on the results of these field tests, we recommend that localized heating, in-situ baselining, and preconcentration be used when possible. PEVA and PVTD polymers appear to work well for the in-situ baselining and preconcentration procedures, respectively, but additional polymers may be suitable as well. The transient processes in the in-situ baselining and preconcentration methods have been shown to overcome the potential problem of drift when using polymer-based sensors. In addition, the remote operation and control of these methods using a telephone line, modem, and commercial data logger provided efficient data collection and ease of use. This system could also be used to post near-real-time data to a web site, if desired (see, for example, www.sandia.gov/sensor/cwl).

Based on these field tests, the use of chemiresistor sensors for monitoring trends and changes in high VOC-concentration environments appears feasible. However, the long-term use and operation of these systems still needs to be tested.

6. References


7. Appendices

7.1 Appendix A: Campbell Scientific CR10X Data-Logging Program for Localized Heating and In-Situ Baselining

;{CR10X}
;
;******************************************************
; This program collects data from sensors deployed at Edwards AFB. The
; sensors include two chemiresistors (E23 and E40),
; thermocouples (one in the groundwater
; and two attached to separate water
; bubblers for in-situ baselining), and a
; water-level pressure transducer.
; The program incorporates multivariate
; regression models developed in
; Statistica 6. In addition, an in-situ
; baselining procedure is controlled
; by this program. When the appropriate
; flag is set high by the user, the
; program will turn on a relay that
; controls pumps that feed air through
; a water bubbler and across the face of
; the sensor via 1/8th-inch tubing.
; The pumps will only turn on when the
; temperature of the bubbler is
; sufficiently high to provide saturated
; water vapor (at the temperature of
; the groundwater) to the sensor.
;
; Lucas K. McGrath and Clifford K. Ho
; Contact: Cliff Ho, 505-844-2384,
; caho@sandia.gov
; 12/5/03
;******************************************************

*Table 1 Program
01: 15 Execution Interval (seconds)

;Flag 1 --> User is ready for a In-Situ Baseline to be calculated if set high
;Flag 2 --> If flag is high the temperature of the bubblers are higher
; then InSitu conditions.
; Note: This has no effect on other aspects of the program.
;Flag 3 --> Tells the program that it is
; between 8 am and 3 pm
; Note: Flag only goes high at
; 8 am exactly and low at 3pm exactly.

;Flag 4 --> If flag is high the Saturated
; Vapor Pressure of the bubblers is
; sufficient to meet the saturated
; vapor pressure of the well
; assuming that humidity from the bubblers
; is 90%
;Flag 7 --> Turns on the baseline
; procedure regardless of temperature of
; bubblers.
;Flag 8 --> If set high it will turn on
; Control port 4
;Port 4 --> Turns on the bubbler pumps

;Hardware ID numbers and explanation of contents.
;001 = Regular monitoring
;002 = Baseline values
;003 = Response of the chemiresistor to
; the pump being turned on.
;004 = Chemiresistor resistances that the
; average is taken of for the baseline.

;RTD calib for E23 Temperature C=1.2663x-245.27
;RTD calib for E40 Temperature C=1.6441x-338.49

;The following instruction reads the
; battery voltage

1:  Batt Voltage (P10)
1: 128 Loc [ Vbatt ]

;The following instructions read the
; chemiresistors, thermocouple, and
; submersible pressure transducer

;The following instruction turns the
; multiplexer on.

2:  Do (P86)
1:  42 Set Port 2 High

3:  Do (P86)
1:  3 Call Subroutine 3

;The following instruction turns off the
; multiplexer
4: Do (P86)
1: 52       Set Port 2 Low

; The following instructions check the
temperature of the well is lower than the
bubblers.
TempDiff1 = BubbTemp1 - WellTemp
TempDiff2 = BubbTemp2 - WellTemp

5: If (X<=>F) (P89)
1: 125      X Loc [ TEMPDIFF1 ]
2: 3        >=
3: 0.0      F
4: 30       Then Do

6: If (X<=>F) (P89)
1: 126      X Loc [ TEMPDIFF2 ]
2: 3        >=
3: 0        F
4: 12       Set Flag 2 High
7: End (P95)

8: If (X<=>F) (P89)
1: 125      X Loc [ TEMPDIFF1 ]
2: 4        <
3: 0.0      F
4: 22       Set Flag 2 Low
9: If (X<=>F) (P89)
1: 126      X Loc [ TEMPDIFF2 ]
2: 4        <
3: 0.0      F
4: 22       Set Flag 2 Low

10: Beginning of Loop (P87)
1: 0        Delay
2: 3        Loop Count
11: Saturation Vapor Pressure (P56)
1: 14      -- Temperature Loc [ WellTemp ]
2: 46      -- Loc [ SatVpWell ]
12: End (P95)

13: If (X<=>Y) (P88)
1: 47       X Loc [ SatVpBub1 ]
2: 3        >=
3: 129      Y Loc [ SVpBub190 ]
4: 30       Then Do

14: If (X<=>Y) (P88)
1: 48       X Loc [ SatVpBub2 ]
2: 3        >=
3: 129      Y Loc [ SVpBub190 ]
4: 14       Set Flag 4 High

15: End (P95)

16: If (X<=>Y) (P88)
1: 47       X Loc [ SatVpBub1 ]
2: 4        <
3: 129      Y Loc [ SVpBub190 ]
4: 24       Set Flag 4 Low

17: If (X<=>Y) (P88)
1: 48       X Loc [ SatVpBub2 ]
2: 4        <
3: 129      Y Loc [ SVpBub190 ]
4: 24       Set Flag 4 Low

18: If time is (P92)
1: 900      Minutes (Seconds --) into a
2: 1440     Interval (same units as
3: 30       above)

19: Z=F x 10^n (P30)
1: 0        F
2: 0        n, Exponent of 10
3: 127      Z Loc [ Counter ]

20: End (P95)

21: If Flag/Port (P91)
1: 11       Do if Flag 1 is High
2: 30       Then Do

SVpBub190 = SatVpWell/0.9

; The following instructions set the
counter to zero at 3 pm to prepare for a
new count the next day.

; The following expressions calculate the
necessary saturated vapor pressure of the
water; bubblers to deliver 100% Rh to the
sensor at the temperature of the
groundwater.
; The relative humidity of the effluent
of the water bubblers is conservatively
assumed to
; be 90% (based on lab tests, which
showed about 95% Rh of the effluent).
If Flag/Port (P91)
1: 14       Do if Flag 4 is High
2: 18       Set Flag 8 High

If Flag/Port (P91)
1: 18       Do if Flag 8 is High
2: 44       Set Port 4 High

End (P95)

;The following instructions record the response of the chemeristor once the pump is turned on.

If Flag/Port (P91)
1: 13       Do if Flag 3 is High
2: 30       Then Do

If Flag/Port (P91)
1: 18       Do if Flag 8 is High
2: 30       Then Do

;The following loop instruction is set to record the response of the chemiresistor to the pump for 30 minutes

Beginning of Loop (P87)
1: 1        Delay
2: 120      Loop Count

;The following instruction turns the multiplexer on.

Do (P86)
1: 42       Set Port 2 High

Do (P86)
1: 3        Call Subroutine 3

;This instruction turns off the multiplexer

Do (P86)
1: 12       Set Port 2 Low

;Reads Battery Voltage during baseline procedure

Batt Voltage (P10)
1: 128      Loc [ Vbatt ]

;The following instructions sample the response of the chemiresistor to the bubbler.

Do (P86)
1: 10       Set Output Flag High (Flag 0)

Set Active Storage Area (P80)^19486
1: 1        Final Storage Area 1

2: 003       Array ID

Resolution (P78)
1: 01       High Resolution

Real Time (P77)^23466
1: 1221     Year,Day,Hour/Minute,Seconds
            (midnight = 2400)

Sample (P70)^6421
1: 16       Reps
2: 1        Loc [ E40RTDOhm ]

Sample (P70)^28016
1: 1        Reps
2: 128      Loc [ Vbatt ]

;The following instruction calculates a baseline via subroutine 2 after the program exits the loop.

Do (P86)
1: 2        Call Subroutine 2

End (P95)

End (P95)

;The following instructions calculate a baseline regardless of flags.
;The following instructions record the response of the chemeristor once the pump is turned on.

If Flag/Port (P91)
1: 17       Do if Flag 7 is High
2: 30       Then Do

Do (P86)
1: 14        Set Port 4 High

Do (P86)
1: 44       Set Port 4 High

;The following loop instruction is set to record the response of the chemiresistor to the pump for 30 minutes

Beginning of Loop (P87)
1: 1        Delay
2: 120      Loop Count

;The following instruction turns the multiplexer on.

Do (P86)
1: 42       Set Port 2 High

Do (P86)
1: 3        Call Subroutine 3

43
;This instruction turns off the multiplexer
47: Do (P86)
1: 12       Set Port 2 Low
;Read battery voltage

48: Batt Voltage (P10)
1: 128      Loc [ Vbatt ]

;The following instructions sample the response of the chemiresistor to the bubbler.
49: Do (P86)
1: 10       Set Output Flag High (Flag 0)

50: Set Active Storage Area (P80)^20773
1: 1        Final Storage Area 1
2: 003      Array ID

51: Resolution (P78)
1: 01       High Resolution

52: Real Time (P77)^23466
1: 1221     Year,Day,Hour/Minute,Seconds
(midnight = 2400)

53: Sample (P70)^6421
1: 16       Reps
2: 1        Loc [ E40RTDOhm ]

54: Sample (P70)^22924
1: 128      Loc [ Vbatt ]

55: End (P95)

;The following instruction calculates a baseline via subroutine 2 after the program exits the loop.
56: Do (P86)
1: 2        Call Subroutine 2

;The following instructions calculate a delta R/Rb for each sensor.
60: If time is (P92)
1: 900      Minutes (Seconds --) into a interval (same units as above)
2: 1440     Interval (same units as above)
3: 23       Set Flag 3 Low

61: If (X<=F) (P89)
1: 20       X Loc [ E40PECH_B ]
2: 3        >=
3: 0.1      F
4: 30       Then Do

62: Beginning of Loop (P87)
1: 0        Delay
2: 8        Loop Count

63: Z=X-Y (P35)
1: 3        X Loc [ E40PECH ]
2: 20       Y Loc [ E40PECH_B ]
3: 30       Z Loc [ NumE40_1 ]

64: Z=X/Y (P38)
1: 30       X Loc [ NumE40_1 ]
2: 20       Y Loc [ E40PECH_B ]
3: 38       Z Loc [ E40drPECH ]

65: End (P95)

66: End (P95)

;The following instructions turn negative delta R/Rb values to zero to apply the calibration
67: Beginning of Loop (P87)
1: 0        Delay
2: 8        Loop Count

68: If (X<=F) (P89)
1: 38       X Loc [ E40drPECH ]
2: 4        <
3: 0.0      F
4: 30       Then Do

69: Z=F x 10^n (P30)
1: 0.0      F
2: 00       n, Exponent of 10
3: 38       Z Loc [ E40drPECH ]

70: End (P95)
The following instructions apply the calibration model for each sensor.

The following equation applies the model for E23 PVT1;
The calibration has an R^2 value of 0.998

\[ \text{PVT1}_{\text{TCE}} = 4.159 + 3.064e7 \times \text{E23dPVT1} - 1.005e6 \times \text{E23dPVT1} \times \text{E23TempC} \]

The following equation applies the model for E23 PVT2;
The calibration has an R^2 value of 0.996

\[ \text{PVT2}_{\text{TCE}} = 4.650e4 + 3.981e5 \times \text{E23dPVT2} - 1.549e3 \times \text{E23TempC} \]

The following equation applies the model for E23 PEVA1;
The calibration has an R^2 value of 0.992

\[ \text{PEVA1}_{\text{TCE}} = 6.330e4 + 7.984e5 \times \text{E23dPEVA1} - 2.108e3 \times \text{E23TempC} \]

The following equation applies the model for E23 PEVA2;
The calibration has an R^2 value of 0.999

\[ \text{PEVA2}_{\text{TCE}} = 3.960 + 2.992e7 \times \text{E23dPEVA2} - 9.845e5 \times \text{E23dPEVA2} \times \text{E23TempC} \]

The following equation applies the model for E23 that includes all polymers;
The calibration has an R^2 value of 0.9997

\[ \text{E23TCEppm} = -1.611 + 3.718e7 \times \text{E23dPVT1} + 4.570e5 \times \text{E23dPEVA2} - 2.502e7 \times \text{E23dPVT2} \times \text{E23dPEVA1} - 1.237e6 \times \text{E23dPVT1} \times \text{E23TempC} - 6.696e3 \times \text{E23dPEVA1} \times \text{E23TempC} \]

The following equation applies the model for E40 PIB;
The calibration has an R^2 value of 0.993

\[ \text{PIB}_{\text{TCE40}} = 1.803e5 + 9.817e5 \times \text{E40drPECH} - 6.002e3 \times \text{E40TempC} \]

The following equation applies the model for E40 PEVA;
The calibration has an R^2 value of 0.999

\[ \text{PEVATCE40} = 4.544e4 - 1.960e6 \times \text{E40drPEVA} - 1.513e3 \times \text{E40TempC} + 6.757e4 \times \text{E40drPEVA} \times \text{E40TempC} \]

The following equation applies the model for E40 PIB and PEVA;
The calibration has an R^2 value of 0.999

\[ \text{PIBPEVATC} = 2.795e4 + 2.541e5 \times \text{E40drPIB} - 9.304e2 \times \text{E40TempC} - 4.471e7 \times \text{E40drPIB} \times \text{E40drPEVA} + 2.429e3 \times \text{E40drPEVA} \times \text{E40TempC} \]

The following instructions sample the desired data.

If time is

1. 0 Minutes (Seconds --) into a
2. 60 Interval (same units as above)
3. 10 Set Output Flag High (Flag 0)

Set Active Storage Area

1. 1 Final Storage Area 1
2. 001 Array ID

Resolution

1. 1 High Resolution

Real Time

1. 1221 Year,Day,Hour/Minute,Seconds (midnight = 2400)

Sample

1. 17 Reps
2. 1 Loc [ E40RTDOhm ]

1. 8 Reps
2. 38 Loc [ E40drPECH ]

1. 5 Reps
2. 115 Loc [ PVT1_{\text{TCE}} ]

1. 6 Reps
2. 121 Loc [ PIBTCE40 ]

1. 2 Reps
2. 131 Loc [ E40Heat_V ]

1. 1 Reps
2. 128 Loc [ Vbatt ]

Sample (P70)^2454
1: 3 Reps  
2: 135 Loc [ PSIg ]  

*Table 2 Program  
02: 0.0000 Execution Interval (seconds)  

;This Table 2 program is for the PreConcentrator  

*Table 3 Subroutines  
1: 1 Subroutine 1  
2: Do (P86)  
1: 73 Pulse Port 3  
3: 1 Ex Channel  
2: 0 Delay W/Ex (0.01 sec units)  
3: 1 Delay After Ex (0.01 sec units)  
4: 0 mV Excitation  
4: End (P95)  

5: 1 Subroutine 2  
5: The following instructions calculate a baseline  
6: Beginning of Loop (P87)  
1: 1 Delay  
2: 8 Loop Count  

;The following instruction turns the multiplexer on.  
7: Do (P86)  
1: 42 Set Port 2 High  

;The following instructions read the RTD on E40.  
8: Do (P86)  
1: 1 Call Subroutine 1  
9: Full Bridge w/mv Excit (P9)  
1: 1 Reps  
2: 14 250 mV Fast Ex Range  
3: 14 250 mV Fast Br Range  
4: 5 DIFF Channel  
5: 3 Excite all reps w/Exchan 3  
6: 250 mV Excitation  
7: 1 Loc [ E40RTDOhm ]  
8: 260.29 Mult  
9: 0.0 Offset  

E40TempC = E40RTDOhm*1.6441 - 338.49  

10: Do (P86)  
1: 1 Call Subroutine 1

11: Excite-Delay (SE) (P4)  
1: 1 Reps  
2: 15 2500 mV Fast Range  
3: 9 SE Channel  
4: 2 Excite all reps w/Exchan 2  
5: 0 Delay (0.01 sec units)  
6: 2500 mV Excitation  
7: 51 -- Loc [ E40PECH_1 ]  
8: 0.0004 Mult  
9: 0.0 Offset  

12: BR Transform Rf[X/(1-X)] (P59)  
1: 1 Reps  
2: 51 -- Loc [ E40PECH_1 ]  
3: 679.4 Multiplier (Rf)  

13: Excite-Delay (SE) (P4)  
1: 1 Reps  
2: 15 2500 mV Fast Range  
3: 10 SE Channel  
4: 2 Excite all reps w/Exchan 2  
5: 0 Delay (0.01 sec units)  
6: 2500 mV Excitation  
7: 59 -- Loc [ E40PNVP_1 ]  
8: 0.0004 Mult  
9: 0.0 Offset  

14: BR Transform Rf[X/(1-X)] (P59)  
1: 1 Reps  
2: 59 -- Loc [ E40PNVP_1 ]  
3: 383.44 Multiplier (Rf)  

15: Excite-Delay (SE) (P4)  
1: 1 Reps  
2: 15 2500 mV Fast Range  
3: 11 SE Channel  
4: 2 Excite all reps w/Exchan 2  
5: 0 Delay (0.01 sec units)  
6: 2500 mV Excitation  
7: 67 -- Loc [ E40PIB_1 ]  
8: 0.0004 Mult  
9: 0.0 Offset  

16: BR Transform Rf[X/(1-X)] (P59)  
1: 1 Reps  
2: 67 -- Loc [ E40PIB_1 ]  
3: 299.66 Multiplier (Rf)  

17: Excite-Delay (SE) (P4)  
1: 1 Reps  
2: 15 2500 mV Fast Range  
3: 12 SE Channel  
4: 2 Excite all reps w/Exchan 2  
5: 0 Delay (0.01 sec units)  
6: 2500 mV Excitation  
7: 75 -- Loc [ E40PEVA_1 ]
The following instructions read the RTD on E23

19: Do (P86)
   1: 1        Call Subroutine 1

20: Full Bridge w/mv Excit (P9)
   1: 1        Reps
   2: 14       250 mV Fast Ex Range
   3: 14       250 mV Fast Br Range
   4: 5        DIFF Channel
   5: 3        Excite all reps w/Exchan 3
   6: 250      mV Excitation
   7: 2        Loc [ E23RTDohm ]
   8: 260.5    Mult
   9: 0.0      Offset

E23TempC = E23RTDohm*1.2663 - 245.27

21: Do (P86)
   1: 1        Call Subroutine 1

22: Excite-Delay (SE) (P4)
   1: 1        Reps
   2: 15       2500 mV Fast Range
   3: 9        SE Channel
   4: 2        Excite all reps w/Exchan 2
   5: 0        Delay (0.01 sec units)
   6: 2500     mV Excitation
   7: 83       -- Loc [ 23PVTD1_1 ]
   8: .0004    Mult
   9: 0.0      Offset

23: BR Transform Rf[X/(1-X)] (P59)
   1: 1        Reps
   2: 83       -- Loc [ 23PVTD1_1 ]
   3: 510.5    Multiplier (Rf)

24: Excite-Delay (SE) (P4)
   1: 1        Reps
   2: 15       2500 mV Fast Range
   3: 10       SE Channel
   4: 2        Excite all reps w/Exchan 2
   5: 0        Delay (0.01 sec units)
   6: 2500     mV Excitation
   7: 91       -- Loc [ 23PVTD2_1 ]
   8: .0004    Mult
   9: 0.0      Offset

25: BR Transform Rf[X/(1-X)] (P59)
   1: 1        Reps
   2: 91       -- Loc [ 23PVTD2_1 ]

26: Excite-Delay (SE) (P4)
   1: 1        Reps
   2: 15       2500 mV Fast Range
   3: 11       SE Channel
   4: 2        Excite all reps w/Exchan 2
   5: 0        Delay (0.01 sec units)
   6: 2500     mV Excitation
   7: 99       -- Loc [ 23PEVA1_1 ]
   8: .0004    Mult
   9: 0.0      Offset

27: BR Transform Rf[X/(1-X)] (P59)
   1: 1        Reps
   2: 99       -- Loc [ 23PEVA1_1 ]
   3: 149.6    Multiplier (Rf)

28: Excite-Delay (SE) (P4)
   1: 1        Reps
   2: 15       2500 mV Fast Range
   3: 12       SE Channel
   4: 2        Excite all reps w/Exchan 2
   5: 0        Delay (0.01 sec units)
   6: 2500     mV Excitation
   7: 107      -- Loc [ 23PEVA2_1 ]
   8: .0004    Mult
   9: 0.0      Offset

29: BR Transform Rf[X/(1-X)] (P59)
   1: 1        Reps
   2: 107      -- Loc [ 23PEVA2_1 ]
   3: 300.4    Multiplier (Rf)

30: Do (P86)
   1: 52       Set Port 2 Low
   31: End (P95)

32: Running Average (P52)
   1: 2        Reps
   2: 11       First Source Loc [ E40TEMPC ]
   3: 28       First Destination Loc [ E40TEMPCB ]
   4: 8        Number of Values in Avg Window

33: Spatial Average (P51)
   1: 8        Swath
   2: 51       First Loc [ E40PECH_1 ]
   3: 20       Avg Loc [ E40PECH_B ]

34: Spatial Average (P51)
   1: 8        Swath
   2: 59       First Loc [ E40PNVP_1 ]
   3: 21       Avg Loc [ E40PNVP_B ]

35: Spatial Average (P51)
   1: 8        Swath
   2: 67       First Loc [ E40PIB_1 ]
   3: 22       Avg Loc [ E40PIB_B ]
Spatial Average (P51)
1: 8 Swath
2: 75 First Loc [E40PEVA_1]
3: 23 Avg Loc [E40PEVA_B]

Spatial Average (P51)
1: 8 Swath
2: 83 First Loc [23PVTD1_1]
3: 24 Avg Loc [E23PVTD1B]

Spatial Average (P51)
1: 8 Swath
2: 91 First Loc [23PVTD2_1]
3: 25 Avg Loc [E23PVTD2B]

Spatial Average (P51)
1: 8 Swath
2: 99 First Loc [23PEVA1_1]
3: 26 Avg Loc [E23PEVA1B]

Spatial Average (P51)
1: 8 Swath
2: 107 First Loc [23PEVA2_1]
3: 27 Avg Loc [E23PEVA2B]

;The following instructions record the baseline values

Do (P86)
1: 10 Set Output Flag High (Flag 0)

Set Active Storage Area (P80)^21555
1: 1 Final Storage Area 1
2: 002 Array ID

Resolution (P78)
1: 1 High Resolution

Real Time (P77)^15612
1: 1221 Year,Day,Hour/Minute,Seconds (midnight = 2400)

Sample (P70)^11095
1: 10 Reps
2: 20 Loc [E40PECH_B]

Do (P86)
1: 10 Set Output Flag High (Flag 0)

Set Active Storage Area (P80)^29668
1: 1 Final Storage Area 1
2: 004 Array ID

Resolution (P78)
1: 1 High Resolution

Sample (P70)^5560
1: 63 Reps
2: 51 Loc [E40PECH_1]

;The following instructions turn off the multiplexer, pump, and baseline flag.

Do (P86)
1: 21 Set Flag 1 Low
2: 28 Set Flag 8 Low

Do (P86)
1: 24 Set Flag 4 Low
2: 54 Set Port 4 Low

Do (P86)
1: 52 Set Port 2 Low

End (P95)

Beginning of Subroutine (P85)
1: 3 Subroutine 3

;The following instructions read the RTD on E40.

Do (P86)
1: 1 Call Subroutine 1

Full Bridge w/mv Excit (P9)
1: 1 Reps
2: 14 250 mV Fast Ex Range
3: 14 250 mV Fast Br Range
4: 5 DIFF Channel
5: 3 Excite all reps w/Exchan 3
6: 250 mV Excitation
7: 1 Loc [E40RTDOhm]
8: 260.29 Mult
9: 0.0 Offset

E40TempC = E40RTDohm*1.6441 - 338.49

;The following instructions read the polymers on E40

Do (P86)
1: 1 Call Subroutine 1

Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 9 SE Channel
4: 2 Excite all reps w/Exchan 2
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 3 Loc [E40PECH]
8: .0004 Mult
9: 0.0 Offset

BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 3 Loc [E40PECH]
3: 679.4 Multiplier (Rf)  7: 2 Loc [ E23RTDohm ]
62: Excite-Delay (SE) (P4)  8: 260.5 Mult
  1: 1 Reps  9: 0.0 Offset
  2: 15 2500 mV Fast Range
  3: 10 SE Channel
  4: 2 Excite all reps w/Exchan 2
  5: 0 Delay (0.01 sec units)
  6: 2500 mV Excitation
  7: 4 Loc [ E40PNVP ]
  8: .0004 Mult
  9: 0.0 Offset 70: Do (P86)

63: BR Transform Rf[X/(1-X)] (P59)  71: Excite-Delay (SE) (P4)
  1: 1 Reps
  2: 4 Loc [ E40PNVP ]
  3: 383.44 Multiplier (Rf)
  4: 2 Excite all reps w/Exchan 2
  5: 0 Delay (0.01 sec units)
  6: 2500 mV Excitation
  7: 7 Loc [ E23PVTD1 ]
  8: .0004 Mult
  9: 0.0 Offset

64: Excite-Delay (SE) (P4)  72: BR Transform Rf[X/(1-X)] (P59)
  1: 1 Reps
  2: 15 2500 mV Fast Range
  3: 11 SE Channel
  4: 2 Excite all reps w/Exchan 2
  5: 0 Delay (0.01 sec units)
  6: 2500 mV Excitation
  7: 8 Loc [ E23PVTD2 ]
  8: .0004 Mult
  9: 0.0 Offset

67: BR Transform Rf[X/(1-X)] (P59)  75: Excite-Delay (SE) (P4)
  1: 1 Reps
  2: 15 2500 mV Fast Range
  3: 11 SE Channel
  4: 2 Excite all reps w/Exchan 2
  5: 0 Delay (0.01 sec units)
  6: 2500 mV Excitation
  7: 9 Loc [ E23PEVA1 ]
  8: .0004 Mult
  9: 0.0 Offset

68: Do (P86)  76: BR Transform Rf[X/(1-X)] (P59)
  1: 1 Call Subroutine 1

69: Full Bridge w/mv Excit (P9)  77: Excite-Delay (SE) (P4)
  1: 1 Reps
  2: 14 250 mV Fast Ex Range
  3: 14 250 mV Fast Br Range
  4: 5 DIFF Channel
  5: 3 Excite all reps w/Exchan 3
  6: 250 mV Excitation

E23TempC = E23RTDohm*1.2663 - 245.27

;The following instructions read the RTD
on E23

;The following instructions read the
polymers on E23
3: 12  SE Channel
4:  2  Excite all reps w/Exchan 2
5:  0  Delay (0.01 sec units)
6: 2500  mV Excitation
7:  10  Loc [ E23PEVA2 ]
8: 0.004  Mult
9:  0.0  Offset

78: BR Transform Rf[X/(1-X)] (P59)
1:  1  Reps
2:  10  Loc [ E23PEVA2 ]
3: 300.4  Multiplier (Rf)

79: Do (P86)
1:  1  Call Subroutine 1

80: Internal Temperature (P17)
1: 13  Loc [ TempRef ]

81: Thermocouple Temp (DIFF) (P14)
1:  1  Reps
2:  11  2.5 mV Fast Range
3:  5   DIFF Channel
4:  1   Type T (Copper-Constantan)
5: 13   Ref Temp (Deg. C) Loc [ TempRef ]
6: 14   Loc [ WellTemp ]
7:  1.0  Mult
8:  0.0  Offset

82: Thermocouple Temp (DIFF) (P14)
1:  1  Reps
2:  11  2.5 mV Fast Range
3:  6   DIFF Channel
4:  1   Type T (Copper-Constantan)
5: 13   Ref Temp (Deg. C) Loc [ TempRef ]
6: 15   Loc [ BubbTemp1 ]
7:  1.0  Mult
8:  0.0  Offset

83: Do (P86)
1:  1  Call Subroutine 1

84: Thermocouple Temp (DIFF) (P14)
1:  1  Reps
2:  11  2.5 mV Fast Range
3:  5   DIFF Channel
4:  1   Type T (Copper-Constantan)
5: 13   Ref Temp (Deg. C) Loc [ TempRef ]
6: 16   Loc [ BubbTemp2 ]
7:  1.0  Mult
8:  0.0  Offset

85: Volt (SE) (P1)
1:  1  Reps
2:  15  2500 mV Fast Range
3:  11  SE Channel

PSICurren = PSImvolts/133
PSIg = 0.6332*PSICurren - 2.4889

86: Volt (SE) (P1)
1: 1  Reps
2: 15  2500 mV Fast Range
3: 12  SE Channel

87: Do (P86)
1:  1  Call Subroutine 1

88: Volt (SE) (P1)
1:  1  Reps
2:  15  2500 mV Fast Range
3:  9   SE Channel

89: End (P95)

End Program

-Input Locations-
1 E40RTDOhm 1 3 2
2 E23RTDohm 1 1 0
3 E40PECH 1 3 2
4 E40PNVF 1 2 2
5 E40PIB 1 2 2
6 E40PEVA 1 2 2
7 E23PVTDL 1 2 2
8 E23PVTDD 1 2 2
9 E23PEVA1 1 2 2
10 E23PEVA2 1 2 2
11 E40TEMPC 1 3 0
12 E23TEMPC 5 2 1
13 TempRef 9 5 1
14 WellTemp 9 3 1
15 BubbTemp1 9 2 1
16 BubbTemp2 9 2 1
17 TempDiff 9 1 1
18 CSI_R 9 0 1
19 CSI_1 9 0 1
20 E40PECH_B 9 4 1
21 E40PNVP_B 9 1 1
22 E40PIB_B 9 1 1
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Appendix B: Campbell Scientific CR10X Data-Logging Program for Integrated Preconcentration

{(CR10X)
;*******************************************************
;* Program: Testing PC Edwards *
;* Wrote By: Jerome Wright *
;* Purpose: To Test the PC with 10 *
;* Prefire Pulses and one *
;* Concentration Pulse *
;*******************************************************

*Table 1 Program
01: 0.25000 Execution Interval (seconds)
1:  If Flag/Port (P91)
1: 16  Do if Flag 6 is High
2: 30  Then Do

;begin purge with ten pulses 5sec pulse
15sec

2:  Beginning of Loop (P87)
1: 1  Delay
2: 10  Loop Count

3:  Beginning of Loop (P87)
1: 1  Delay
2: 20  Loop Count

4:  Do (P86)
1: 41  Set Port 1 High

5:  Excite-Delay (SE) (P4)
1: 1  Reps
2: 15 2500 mV Fast Range
3: 3  SE Channel
4: 1  Excite all reps w/Exchan 1
5: 0  Delay (0.01 sec units)
6: 2500 mV Excitation
7: 2  Loc [ PCPEVA2 ]
8: .0004  Multi
9: 0.0  Offset

8:  BR Transform Rf[X/(1-X)] (P59)
1: 1  Reps
2: 2  Loc [ PCPEVA2 ]
3: 12030 Multiplier (Rf)

9:  Excite-Delay (SE) (P4)
1: 1  Reps
2: 15 2500 mV Fast Range
3: 3  SE Channel
4: 1  Excite all reps w/Exchan 1
5: 0  Delay (0.01 sec units)
6: 2500 mV Excitation
7: 3  Loc [ PCPEVA3 ]
8: .0004  Multi
9: 0.0  Offset

10:  BR Transform Rf[X/(1-X)] (P59)
1: 1  Reps
2: 3  Loc [ PCPEVA3 ]
3: 56360 Multiplier (Rf)

11:  Excite-Delay (SE) (P4)
1: 1  Reps
2: 15 2500 mV Fast Range
3: 4  SE Channel
4: 1  Excite all reps w/Exchan 1
5: 0  Delay (0.01 sec units)
6: 2500 mV Excitation
7: 4  Loc [ PCPEVA4 ]
8: .0004  Multi
9: 0.0  Offset

PCPEVA4a=PCPEVA4/100

12:  BR Transform Rf[X/(1-X)] (P59)
1: 1  Reps
2: 5  Loc [ PCPEVA4a ]
3: 5100 Multiplier (Rf)

13:  Z=X*F (P37)
1: 5  X Loc [ PCPEVA4a ]
2: 100 F
3: 4  Z Loc [ PCPEVA4 ]

1: 1  Reps

54
14: Do (P86)
1: 10 Set Output Flag High (Flag 0)

15: Set Active Storage Area (P80)^26654
1: 1 Final Storage Area 1
2: 015 Array ID

16: Resolution (P78)
1: 1 High Resolution

17: Real Time (P77)^5377
1: 1221 Year, Day, Hour/Minute, Seconds (midnight = 2400)

18: Sample (P70)^27559
1: 4 Reps
2: 1 Loc [ PCPEVA1 ]

19: End (P95)

20: Do (P86)
1: 51 Set Port 1 Low

21: Beginning of Loop (P87)
1: 1 Delay
2: 60 Loop Count

22: Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 1 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 1 Loc [ PCPEVA1 ]
8: .0004 Mult
9: 0.0 Offset

23: BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 3 Loc [ PCPEVA3 ]
3: 56360 Multiplier (Rf)

24: Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 1 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 1 Loc [ PCPEVA1 ]
8: .0004 Mult
9: 0.0 Offset

25: BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 2 Loc [ PCPEVA2 ]

26: Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 3 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 3 Loc [ PCPEVA3 ]
8: .0004 Mult
9: 0.0 Offset

27: BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 3 Loc [ PCPEVA3 ]
3: 56360 Multiplier (Rf)

28: Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 4 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 4 Loc [ PCPEVA4 ]
8: .0004 Mult
9: 0.0 Offset

29: BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 5 Loc [ PCPEVA4a ]
3: 5100 Multiplier (Rf)

30: Z=X*F (P37)
1: 5 X Loc [ PCPEVA4a ]
2: 100 F
3: 4 Z Loc [ PCPEVA4 ]

31: Do (P86)
1: 10 Set Output Flag High (Flag 0)

32: Set Active Storage Area (P80)^1369
1: 1 Final Storage Area 1
2: 015 Array ID

33: Resolution (P78)
1: 1 High Resolution

34: Real Time (P77)^23061
1: 1221 Year, Day, Hour/Minute, Seconds (midnight = 2400)

35: Sample (P70)^10445
1: 4 Reps
2: 1 Loc [ PCPEVA1 ]

PCPEVA4a=PCPEVA4/100
End (P95)

; Delay time is set to 3530 becuase of a 60 (15 second) of post plus 10 (2.5 seconds) Baseline for the final pulse.

Beginning of Loop (P87)
1: 3530 Delay
2: 1 Loop Count

End (P95)

;Collect Baseline

; Pulse data collection 8 samples/sec for 5 seconds

Beginning of Loop (P87)
1: 1 Delay
2: 20 Loop Count

Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 1 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 1 Loc [ PCPEVA1 ]
8: .0004 Mult
9: 0.0 Offset

BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 3 Loc [ PCPEVA3 ]
3: 56360 Multiplier (Rf)

Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 4 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 4 Loc [ PCPEVA4 ]
8: .0004 Mult
9: 0.0 Offset

BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 3 Loc [ PCPEVA3 ]
3: 56360 Multiplier (Rf)

Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 2 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 2 Loc [ PCPEVA2 ]
8: .0004 Mult
9: 0.0 Offset

BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 2 Loc [ PCPEVA2 ]
3: 12030 Multiplier (Rf)

Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 3 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 3 Loc [ PCPEVA3 ]
8: .0004 Mult
9: 0.0 Offset

BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 3 Loc [ PCPEVA3 ]
3: 56360 Multiplier (Rf)

Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 4 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 4 Loc [ PCPEVA4 ]
8: .0004 Mult
9: 0.0 Offset

BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 3 Loc [ PCPEVA3 ]
3: 56360 Multiplier (Rf)

Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 2 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 2 Loc [ PCPEVA2 ]
8: .0004 Mult
9: 0.0 Offset

BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 2 Loc [ PCPEVA2 ]
3: 12030 Multiplier (Rf)

Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 3 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 3 Loc [ PCPEVA3 ]
8: .0004 Mult
9: 0.0 Offset

BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 3 Loc [ PCPEVA3 ]
3: 56360 Multiplier (Rf)
Pulse data collection 8 samples/sec for 5 seconds

56: Beginning of Loop (P87)
1: 1 Delay
2: 20 Loop Count

57: Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 1 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 1 Loc [ PCPEVA1 ]
8: .0004 Mult
9: 0.0 Offset

64: BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 5 Loc [ PCPEVA4 ]
3: 5100 Multiplier (Rf)

58: BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 1 Loc [ PCPEVA1 ]
3: 4223 Multiplier (Rf)

59: Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 2 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 2 Loc [ PCPEVA2 ]
8: .0004 Mult
9: 0.0 Offset

60: BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 2 Loc [ PCPEVA2 ]
3: 12030 Multiplier (Rf)

61: Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 3 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 3 Loc [ PCPEVA3 ]
8: .0004 Mult
9: 0.0 Offset

62: BR Transform Rf[X/(1-X)] (P59)
1: 1 Reps
2: 3 Loc [ PCPEVA3 ]
3: 56360 Multiplier (Rf)

63: Excite-Delay (SE) (P4)
1: 1 Reps
2: 15 2500 mV Fast Range
3: 4 SE Channel
4: 1 Excite all reps w/Exchan 1
5: 0 Delay (0.01 sec units)
6: 2500 mV Excitation
7: 1 Loc [ PCPEVA1 ]
8: .0004 Mult
PCPEVA4a = PCPEVA4/100

PCPEVA1 1 10 10
PCPEVA2 1 10 10
PCPEVA3 1 10 10
PCPEVA4 1 10 5

CSI_R 0 0 0
CSI_1 0 0 0

X 0 0 0
F 0 0 0
Z 0 0 0
Program Security

Mode 4-
Final Storage Area 2-
CR10X ID-
CR10X Power Up-
CR10X Compile Setting-
CR10X RS-232 Setting-
DLD File Labels-
Final Storage Labels-
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1, Hour_Minute_RTM
1, Seconds_RTM
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2, PCPEVA3-3
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4, Day_RTM
4, Hour_Minute_RTM
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7, Day_RTM
7, Hour_Minute_RTM
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