

PORTABLE POTENTIOSTAT

By

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# ABSTRACT OF THE THESIS

## PORTABLE POTENTIOSTAT

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THESIS DIRECTOR:

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As the world is moving forward trying to improve the purity of water, some parts are still faced with severe water purity crisis. Many drinking water supplies are contaminated by lead traces higher than the permissible limit which is 15 ug/L as decided by the US EPA. This lead contamination mostly occurs due to corrosion or solder joints in pipes, further leading to deaths or carcinogenic implications among victims. Thus to find a cost effective and precise solution, we here present a novel method to detect lead in water sources, with high accuracy using low powered portable device. The circuit consists of a 3-electrode potentiostat later connected to a Trans-Impedance Amplifier, LPF and then the signal is read through a 12-bit ADC. A digital data measurement and analysis logic was designed in LabVIEW, which read the voltammograms plots. We tested our circuit on three different concentration of lead in nitric acid/acetate buffer. The solution is scanned with a linear

wave stripping voltage and the current is measured to analyze peaks at the redox potential of lead. The amplitude of this peak determines the concentration of lead in the solution. Thus we were able to detect distinct peaks for these three concentrations. This palm sized device, designed using low end off the shelf components, will be later used to serve as an input to a lead filtration system which can be installed on site.

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# Chapter 1

## Introduction

### 1.1 Potentiostat

A potentiostat is a control and measuring device. It has an electronic circuit which controls the voltage between two electrodes, i.e. Working Electrode and Reference Electrode which are dipped in an electrochemical cell. This control is implemented by injecting current through a third electrode i.e. Counter Electrode. Current measured on the Working Electrode is later used for analysis. Here we have used it to demonstrate detection of lead concentrations in water and thus further will be used to decide whether it is above the permissible limit decide by the US EPA [3][9].

### 1.2 3-Electrode System

#### Reference Electrode:

This electrode acts as a voltage reference for Working electrode. I.e. the voltage measurements are not w.r.t ground but w.r.t this electrode. Hence it proves to be a critical part of the experiments. It is designed such that, voltage is kept constant and no current flows through it. As this electrode has just one dedicated task, the stability is quite superior and thus 3-electrode systems offers better operation and measurements.

Working Electrode: This electrode is the point of focus in electrochemical experiments. Current is observed by varying the potential and inducing a reaction in the solution of interest over this electrode. Thus, the material used for this electrode is mostly inert to avoid any alterations to the reaction. The working electrode is further connected to a transimpedance amplifier. This converts the measured current to voltage and thus these reading can be filtered transmitted and analyzed.

Counter Electrode: This electrode completes the circuit. The current entering the solution through working electrode leaves it through this electrode. It is mostly made from the working electrode material. The applied voltage is varied through this electrode.

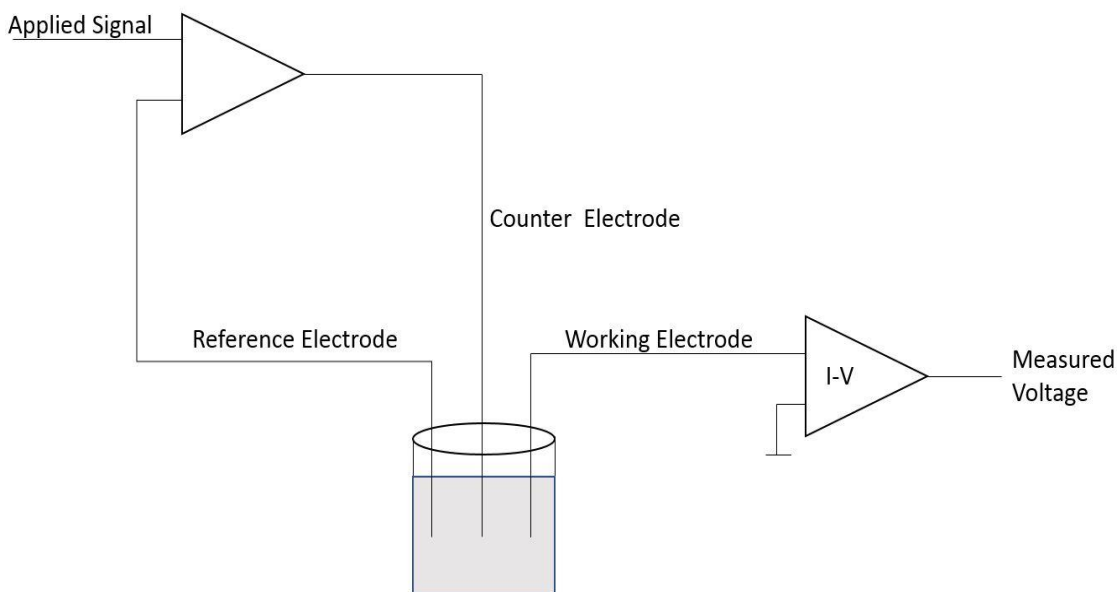


Figure 1.1 3-Electrode Cell

### 1.3 Voltammetry

It is an electroanalytical method used to identify traces of metals in a solution by applying a potential across electrochemical cell and measuring the current. This current is plotted vs. the applied potential, forming a plot known as voltammograms. This plot contains information about the reaction occurred in the solution. The pattern of the applied potential determines the type of voltammetry. Different types of voltammetry are used depending on the analyte to be measured in the solution [10]. Some common types include Cyclic Voltammetry, Linear Voltammetry, differential pulse Voltammetry etc. Here we have used Square wave anodic stripping Voltammetry to detect quantity of lead in the solution.

#### Square wave anodic stripping Voltammetry:

In this technique, the required analyte is first electroplated on the working electrode by applying specific voltage for a specific duration. Then a square wave imposed on a ramp voltage is applied with respect to the reference electrode. The current is measured during this period. The current measured follows that of applied voltage until the oxidation potential, at this peak lead oxidizes and emits electrons thus creating an additional faradaic current. This creates a current peak proportional to the additional electrons [2]. The amplitude of this peak determines the analyte concentration in the solution. After this measurement, a constant potential with opposite polarity is applied, to clean the working electrode from any remaining electroplated electrolyte.



## **1.4 Statement of the problem**

Voltammetry is done using a high end, robust bench top device in industry as well as in research labs however these devices are bulky and requires a large power supply. Also, the device is controlled through a software which provides configurations for a wide variety of Voltammetric experiments most of which are not required for our purpose. Thus, it is difficult to use these devices for on-field applications like ours. Thus, our main aim was to build a robust, miniature portable device which can be installed on-site for lead detection in river water.

## **1.5 Objective and Approach**

For this experiment we needed to detect the current through the working electrode and plot it vs the applied potential to measure the amplitude of the peak at redox potential. This peak will correspond to the concentration of lead in the solution. The main problem was that the current peak detected was of the order of microamps, so we had to build a system to overcome the noise (introduced by the sensor, circuit and the environment) as well as effectively plot the signal. The aim was to work on finding a low power low noise board and modify it according to our requirements.

Thus, we first did a literature review of the existing potentiostats available in the market and then choose the one designed by Kevin Plaxco group at University of California Santa Barbara [1]. We analyzed the circuit first and made a lot of modifications. The

board had power supply limitations, lack of transient protection circuitry, additional lcd & memory modules etc. Also, our sensor was sensitive to voltage and had a maximum allowable voltage of 1v, thus we redesigned the opamp circuit to accommodate our limitations as well. We compiled an embedded C code strictly applicable to our circuit specifications and later burnt it on the microcontroller. We also designed a simple data acquisition LabVIEW code to read and document the data after the experiment.

## **1.6 Organization of Thesis**

In Chapter 2, the boards mixed signal circuitry is discussed. It contains designs of the various blocks like signal generation circuit, Transimpedance amplifier, low pass filter, microcontroller etc. The design equations and transfer function of each block are also presented. It also describes the overall block diagram and noise analysis of the circuit.

The next chapter focuses on Data acquisition and plotting using LabVIEW and specifies in brief the data flow throughout the code. The specifications of the experiment, sample preparation, design of electrodes and results are presented in Chapter 4. Chapter 5 presents the future scope of this projects and suggests improvements over the existing board.

## Chapter 2

### Mixed – Signal Circuitry

#### 2.1 Analog Circuit

Control amplifier: TTL2264ID quad opamp IC is used as an amplifier in the circuit. The signal of interest is supplied to the control amplifier opamp whose output is connected to the counter electrode. The reference electrode is connected to the negative input through a buffer. This negative feedback is used to get a stable desired waveform. Feedback capacitor of 1uf was also used to avoid self-oscillation of the opamp. The voltage applied to the electrodes through the counter electrode is given by:

$$V_{CE} = \left(1 + \frac{R6}{R5}\right) V_+ - \left(\frac{R6}{R5}\right) V_- \dots\dots\dots (2)$$

Where R6 and R5 are the negative feedback resistors and  $V_+$   $V_-$  are the input voltages at the non-inverting and inverting terminals of the opamp respectively.

Transimpedance amplifier (I-V): The same quad opamp IC is used to convert the measured current at working electrode. Appropriate feedback resistor is used to accommodate the expected current levels. We have used a DG612A quad switch to select feedback resistor through software control. The transimpedance amplifier enables the detected signal to be transmitted, filtered or amplified if required.

The output voltage and the transfer function (TF) of the Transimpedance amplifier is given as:

$$V_{out} = -I_{in} \cdot R_f \dots\dots\dots (3)$$

$$TF: \quad \frac{V_{out}}{I_{in}} = \frac{-R_f}{1+s \cdot R_f \cdot C_f} \dots\dots\dots (4)$$

$$F_p = \frac{1}{2 \cdot \pi \cdot R_f \cdot C_f} \dots\dots\dots (5)$$

Where,  $R_f$  and  $C_f$  are the feedback components of the TIA and  $I_{in}$  is the current measured at the Working Electrode.  $F_p$  is the pole frequency after which the gain of the amplifier reduces. This equation was used to determine the value of the feedback capacitor. The bandwidth was decided such that it is much higher than that of the desired current peaks so as to read them effectively without degrading the amplitude [12].

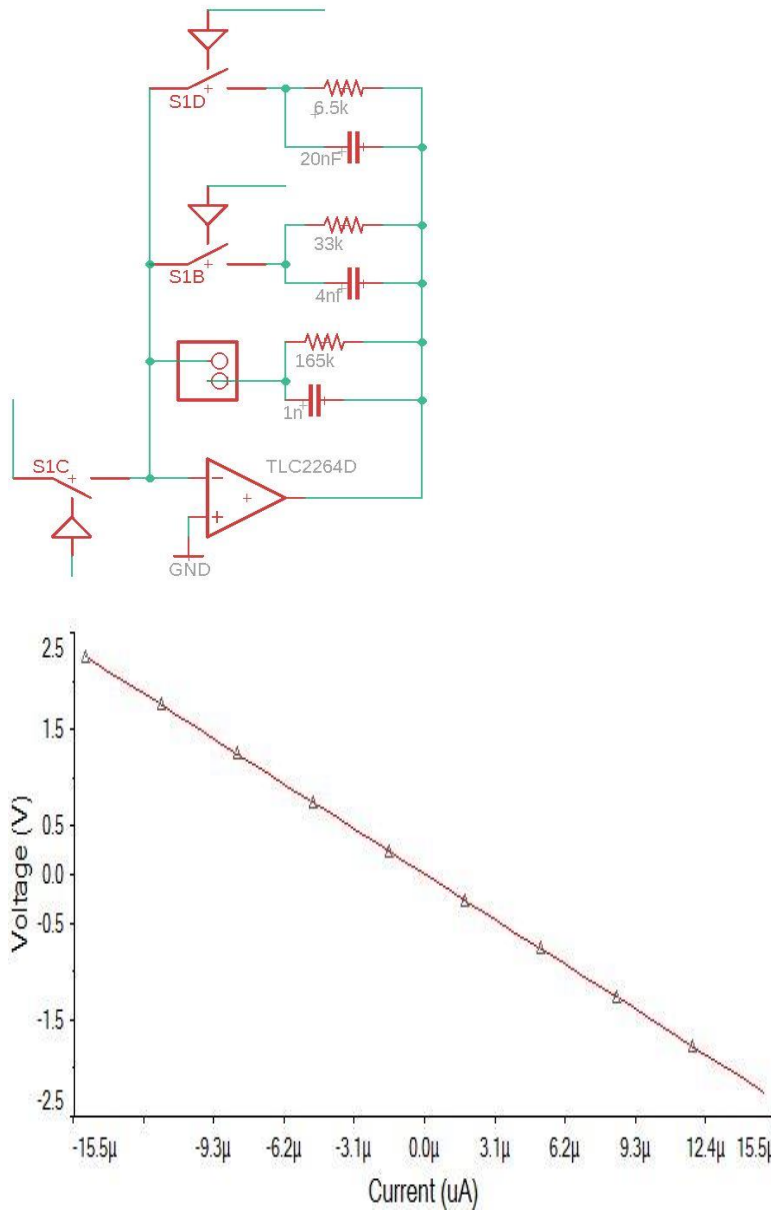


Figure 2.1 Transimpedance amplifier design and simulated response

Low Pass Filter: A 2nd order butter worth filter is designed using the opamp IC. The cutoff is 1Khz as a desirable peak with higher frequency is not expected. A gain configuration was also added to the filter. This can be used for amplification of the measured signal if the desired peaks are not distinct enough. Also embedding the gain feature in the filter itself

avoids the need of additional gain stage which helps in limiting the power consumption as well as the noise [11].

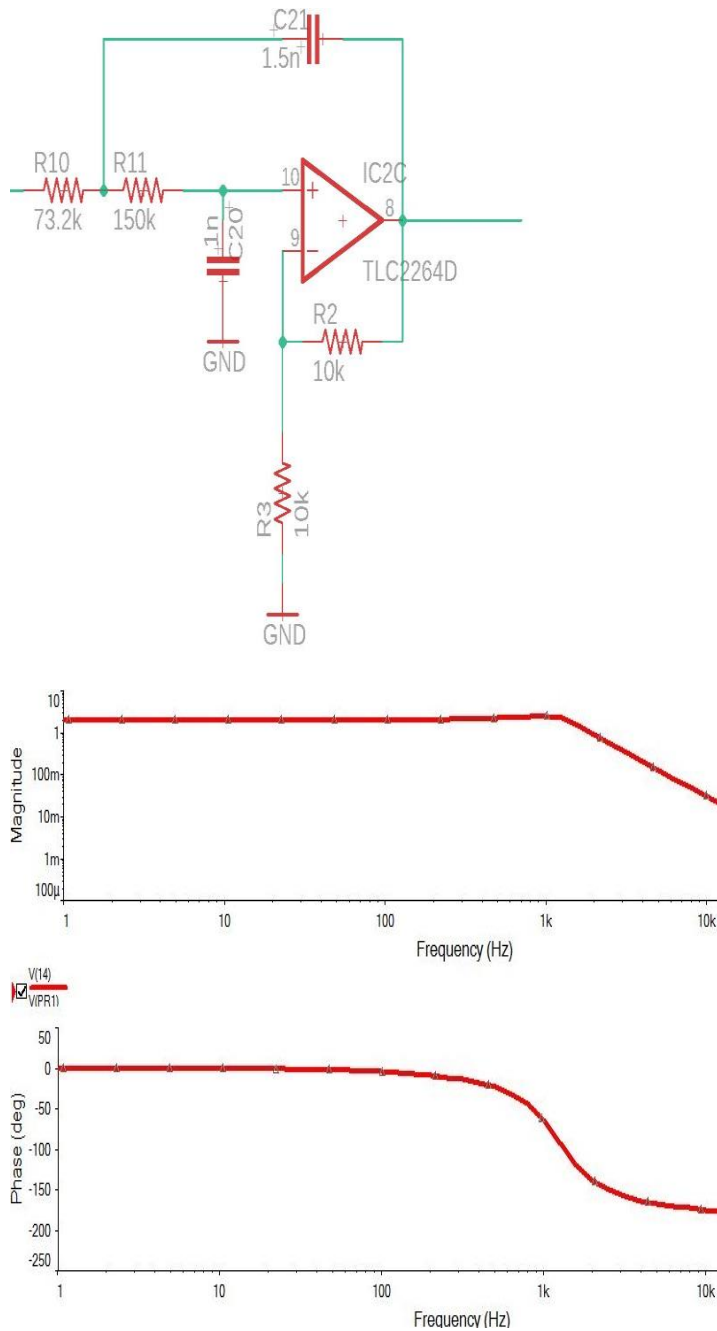


Figure 2.2 Low pass filter Design and simulated response

The cutoff frequency and the gain can be calculated as:

$$F_c = \frac{1}{2\pi\sqrt{R_{10} \cdot R_{11} \cdot C_{20} \cdot C_{21}}} \dots\dots\dots (6)$$

$$Gain(A) = 1 + \frac{R_2}{R_3} \dots\dots\dots (7)$$

The transfer function of the second order Butterworth low pass filter is given as:

$$\frac{V_o}{V_{in}} = \frac{A}{s^2 + \frac{(R_{11} \cdot C_{20} + R_{10} \cdot C_{20} + (1 - A)R_{10} \cdot C_{21})}{R_{10} \cdot R_{11}} s + \frac{1}{R_{10} \cdot R_{11} \cdot C_{20} \cdot C_{21}}} \dots\dots\dots (8)$$

Where the above-mentioned components in equations (6) (7) & (8) can be found in the Figure 2.2

## 2.2 Digital Circuit

Atmega32a4u: This microcontroller of the Atmel family is used to control the board. It is used to generate the signal, store & transmit data and control the feedback resistor switch positions. As the real-world signals are analog while the analyzed data is digital, ADC and DAC embedded in the microcontroller are used for processing [5].

Atmel DAC and ADC: A 12-bit high speed DAC is used to generate the desired signal with a resolution of 0.8mV. Thus, the DAC supports generation of most of the required Voltammetric signals for experiments. The output of the DAC is connected to the positive input of the control amplifier. A SAR 12-bit ADC is used to convert the measured analog voltages to digital values. This helps in data processing and serial data transfer from the board to laptop. The configuration of ADC, DAC and USART was done by writing an embedded C code in Atmel studio and was later burnt on the microcontroller using atmel ICE programmer. The voltage generated by the DAC can be configured as:

$$V_o = \left( \frac{RES}{4095} \right) V_{ref} \dots\dots\dots (9)$$

Where RES is the digital input from the C code to produce the desired voltage and  $V_{ref}$  is the external 3.3v reference voltage provided to the DAC. No gain or offset correction was required for generating or measuring the signal. Based on equation (9), the Resolution of the DAC can be calculated as:

$$Resolution = \frac{V_{ref}}{4095} = \frac{3.3v}{4095} = 0.8mv \dots\dots\dots (10)$$

## 2.3 Power Supply

The power supplied to this board is from 2 sources. The positive supply is obtained through the USB cable which draws power form the connected laptop. This positive voltage is stepped down using FAN2500S33X IC to 3.3 volts with current capacity of 200mA. This IC was used as the required supply voltage specifications by the microcontroller, opamp

and switch IC is met by this IC. The negative power supply is derived from LT 1964 IC. This negative power supply is connected to only to the opamp negative power supply to accommodate negative voltage measured by transimpedance amplifier up to -3.3 volts. The positive and negative power supplies are designed as follows:

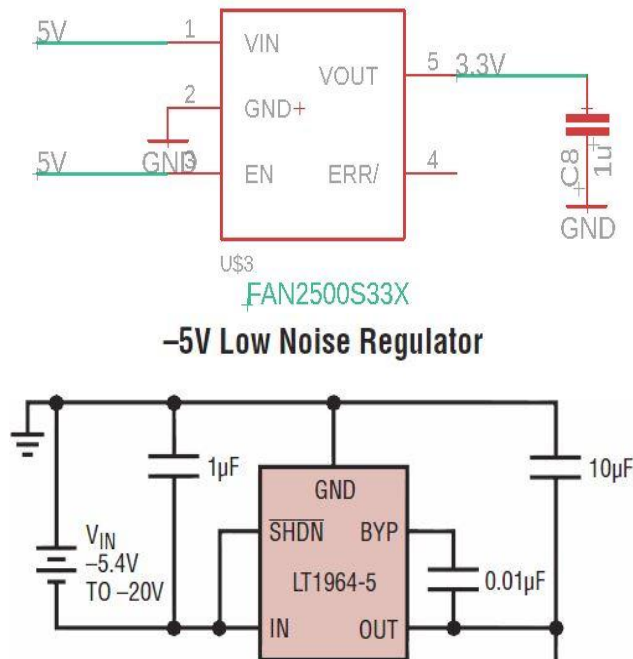


Figure 2.3 Positive and negative Power Supply

## 2.4 Block Diagram and noise analysis

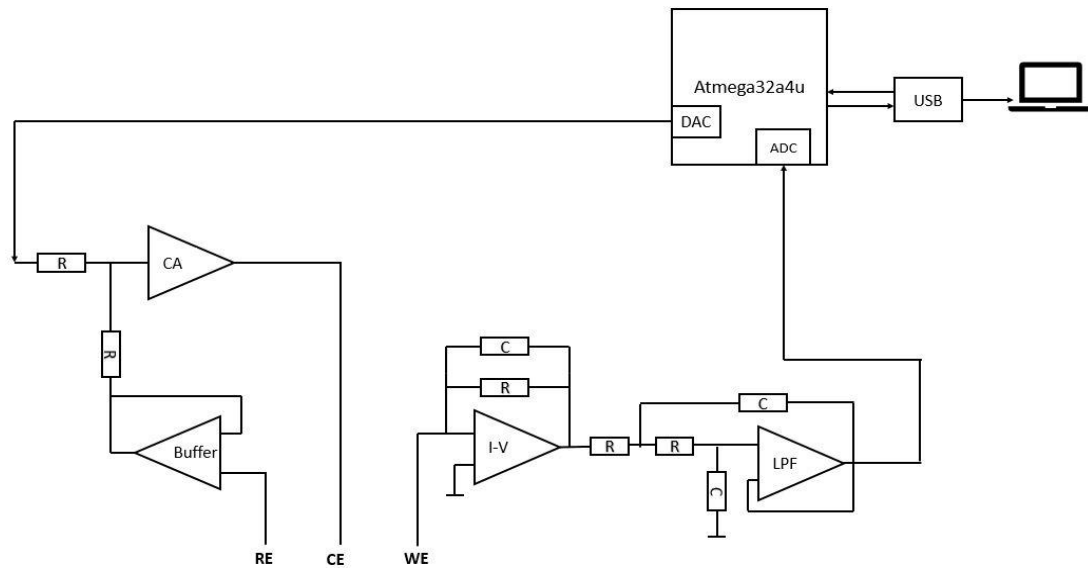


Figure 2.4 Block Diagram

Figure 2.4 shows the complete block diagram of the designed pcb. It consists of the signal flow generated from the microcontroller Digital to Analog Converter (DAC) and then through the Transimpedance amplifier (I-V) and filter back to the atmega ADC. Later this data is sent through an FTDI chip to the USB mini connector and then is analyzed in the PC.

#### Theoretical Noise Model:

Output referred noise of the circuit was calculated by dividing the systems into three blocks. Individual noise of the system was calculated and then the cumulative noise power was calculated. The solution was simulated by replacing the it with two 10kohm resistors. Each connected to Counter Electrode and Working Electrode. The Reference Electrode was connected to the junction of these two resistors. No resistor was connected to the

reference electrode as no current flows through it. The following figure shows the noise model and various noise sources.

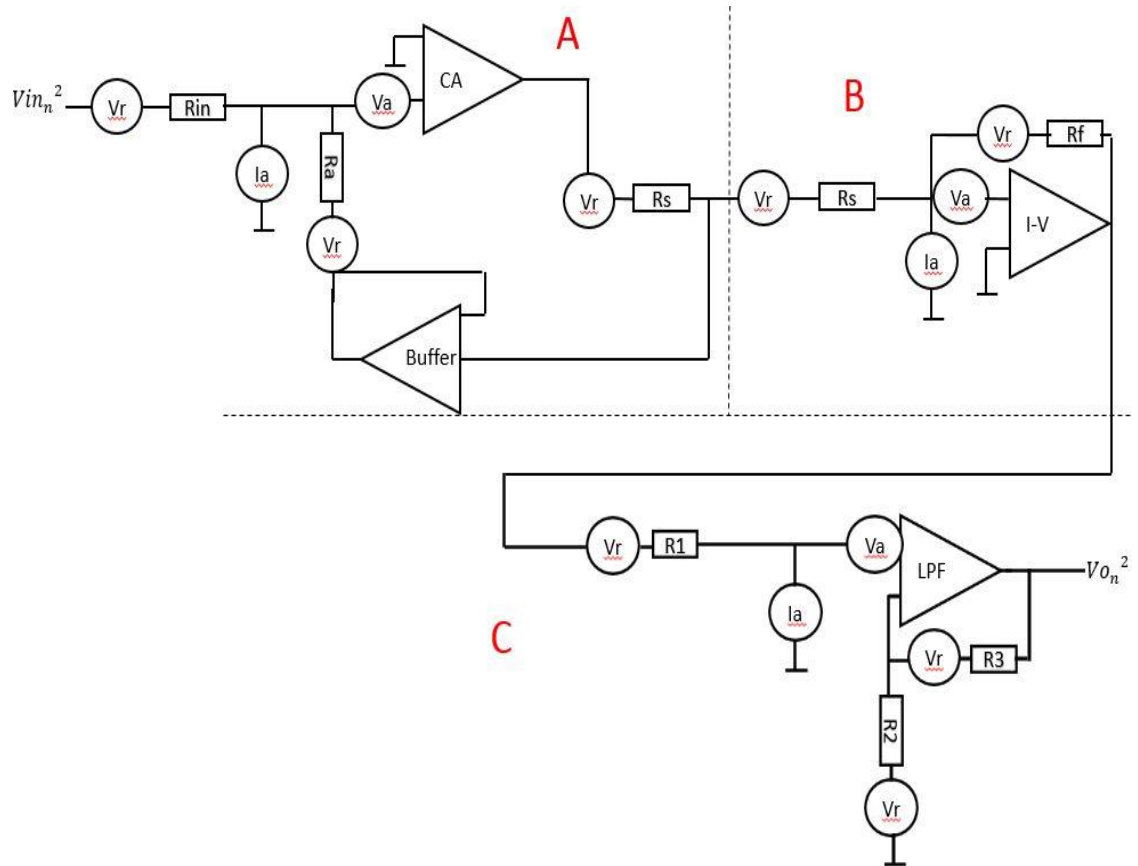


Figure 2.5 Theoretical Noise Model

Input referred noise equation of block A (Control Amplifier):

$$\overline{V_c^2} = \overline{V_{Rin_n}^2} + \overline{V_{A_n}^2} + \frac{\overline{V_{Rcn}^2}}{R_c^2} \cdot Rin^2 + Rin^2 \cdot \overline{I_{A_n}^2} = 5.4 \text{ nV}^2$$

..... (11)

Input referred noise equation of block B (Transimpedance amplifier):

$$\overline{V_T^2} = \overline{V_{Rs_n}^2} + \overline{V_{A_n}^2} + \frac{\overline{V_{Rfn}^2}}{Rf^2} \cdot Rs^2 + Rs^2 \cdot \overline{I_{A_n}^2} =$$

$$0.35 \mu V^2 \dots\dots\dots (12)$$

Input referred noise equation of block C (Low Pass filter):

$$\overline{V_L^2} = \overline{V_{R1_n}^2} + \overline{V_{A_n}^2} + \frac{R2^2}{(R2+R3)^2} \cdot \overline{V_{R3_n}^2} + R1^2 \cdot \overline{I_{A_n}^2} + \frac{R3^2}{(R2+R3)^2} \cdot \overline{V_{R2_n}^2} =$$

$$1.36 \mu V^2 \dots\dots\dots (13)$$

Thus the out referred noise system was calculated by considering voltage gain from each stage as follows:

$$\overline{V_{tot_n}^2} = \left( \left( \overline{V_c^2} \cdot G_c^2 + \overline{V_T^2} \right) \cdot G_T^2 + \overline{V_L^2} \right) \cdot G_L^2 =$$

$$6.96 \mu V^2 \dots\dots\dots (14)$$

Thus the total noise power of the system is calculated to be  $6.96 \mu V^2$  but as this model has considered only limited noise sources the value is so low. We further simulated Noise model of each block in NI Multisim 14.1 with the help of “Noise Analyses and simulation”. Also all the bypass capacitors were included in the simulation circuit. The output referred noise voltage was  $51.6 \mu V^2$ . This simulated value is higher because it considers even more noise sources such as the power supply noise, opamp noise etc. Later we simulated the system block wise to get the noise spectrum. The following figure shows the output noise spectrum of the system from 1Hz to 10 KHz.

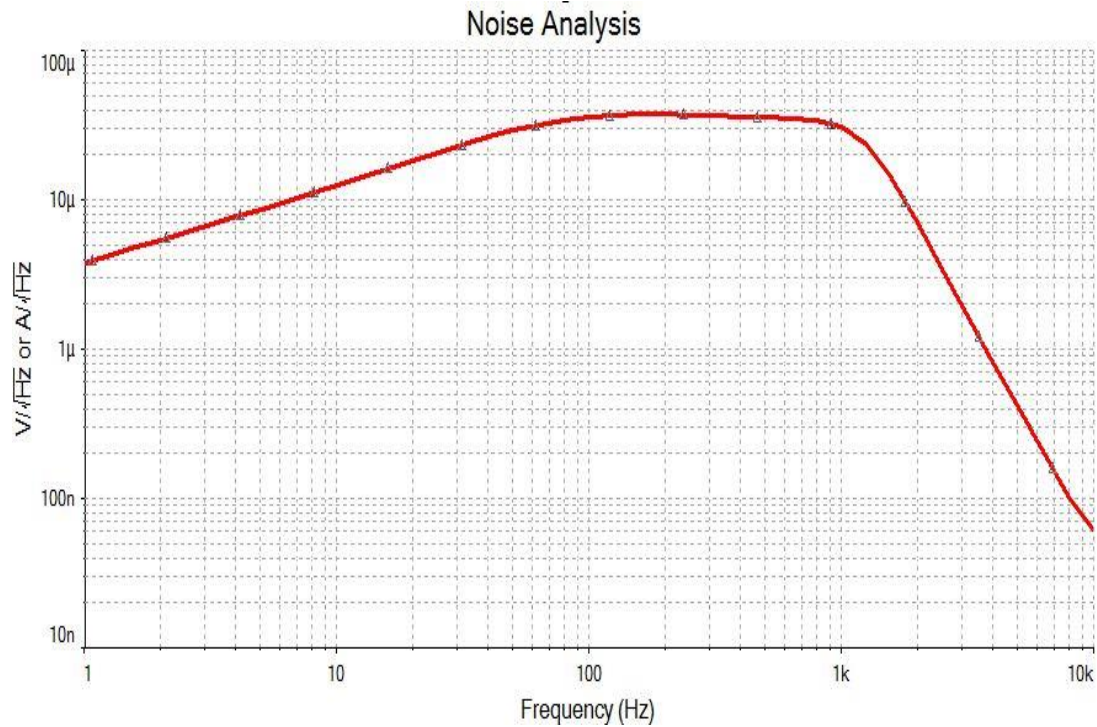


Figure 2.6 Noise Spectrum of the system

The Noise spectrum shows changes in the slope of the curve twice. The wave flattens out after 72.3 Hz as this is the unity gain bandwidth of the Trans impedance amplifier, after which the gain drops, and the noise level becomes constant. This frequency is calculated by the equation (5) mentioned in the Trans impedance amplifier section. The noise drops drastically after 1 KHz because of the low pass filter. The second order Butterworth filter has the cutoff frequency of 1 KHz after which the gain drops at the rate of 40dB per decade and thus the noise level also drops. The simulated rms value of noise is 1.24 mV, however the measured rms value of the board is 70mV. The noise levels in the simulation are very low as compared to that of actual levels because of several factors. Simulation does not include the noise from the microcontroller, ICs, wires and SPE cable, crocodile connector clips, environmental noise, board level noise or stray capacitances etc. Now, we considered the noise level from our board with and without the sensor.

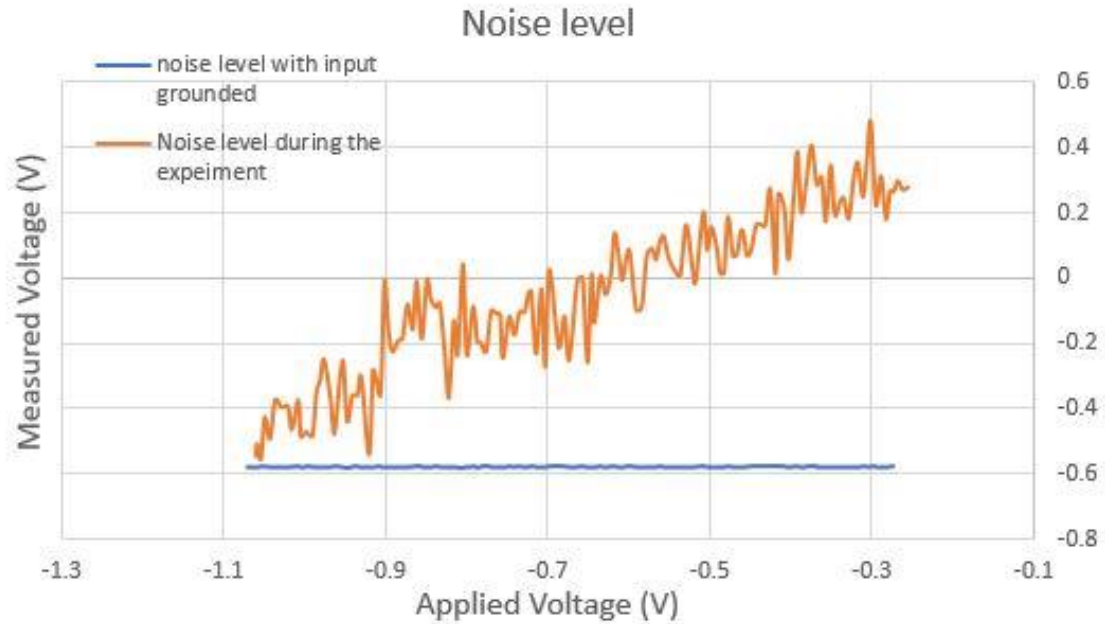


Figure 2.7 Noise Analysis of the circuit

The above figure shows the noise levels for two different settings. It is the plot of measured voltage at the output of low pass filter vs. the applied potential. The blue wave is the measured output when the inputs to the transimpedance amplifier are grounded thus resulting in a constant voltage at the output. As the sensor is not connected the output has very low rms noise in the order of 1-2 mv. The orange wave is the measured output voltage during one of the experiments conducted by connecting the sensor dipped in the solution. This wave has the noise level of 70 mv rms.

## Chapter 3

### Data Acquisition and PCB Design

Atmega 32 USART serial communication module was used to transfer the read data from the experiment to the PC. The data is transferred in bulk after the experiment is complete. It fetches data from the ADC registers of the microcontroller. FT232 USART to USB interface was used to transfer the data from the atmega USART to the USB mini connector. The transferred data was read by Labview code in the laptop[13].

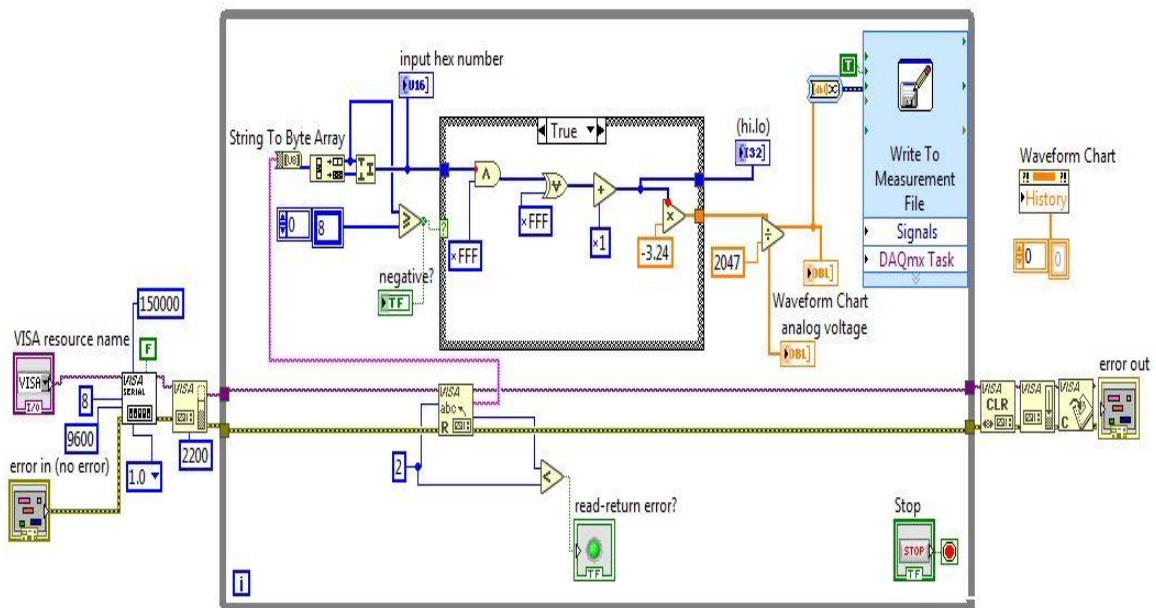


Figure 3.1 Labview Code for Data acquisition

The LabVIEW code reads and plots the data continuously. The hex data read from the board is converted to digital for processing. This digital data is converted to analog using a DAC designed in the code itself. The DAC is configured using the “result representation table” which is specified in the Atmega A manual. Thus, after converting the data, it is simultaneously plotted as well as written to an MS-excel file. The specific Voltammetric graphs are then plotted and analyzed in MS-excel.

The atmega ADC is configured to read voltages in the range  $-V_{ref}$  to  $V_{ref}$ . I.e.-2047 represents  $-V_{ref}$  and 2048 represent  $+V_{ref}$ . Hence each read value is represented by 11 bits and accordingly appropriate LabVIEW DAC is designed to mimic the role as that of atmega ADC.

$$V_o = +/ - \frac{RES}{2048} \cdot V_{ref} \dots\dots\dots (15)$$

## Chapter 4

### Measurement and Result Analysis

#### 4.1 Experiment Setup

The board was connected to an SPE cable through alligator clips. The other end of the SPE cable houses the screen-printed electrode. The Screen-printed electrode is dipped in the solution while the board continuously read the voltages through this cable. The following image shows the setup of the experiment.

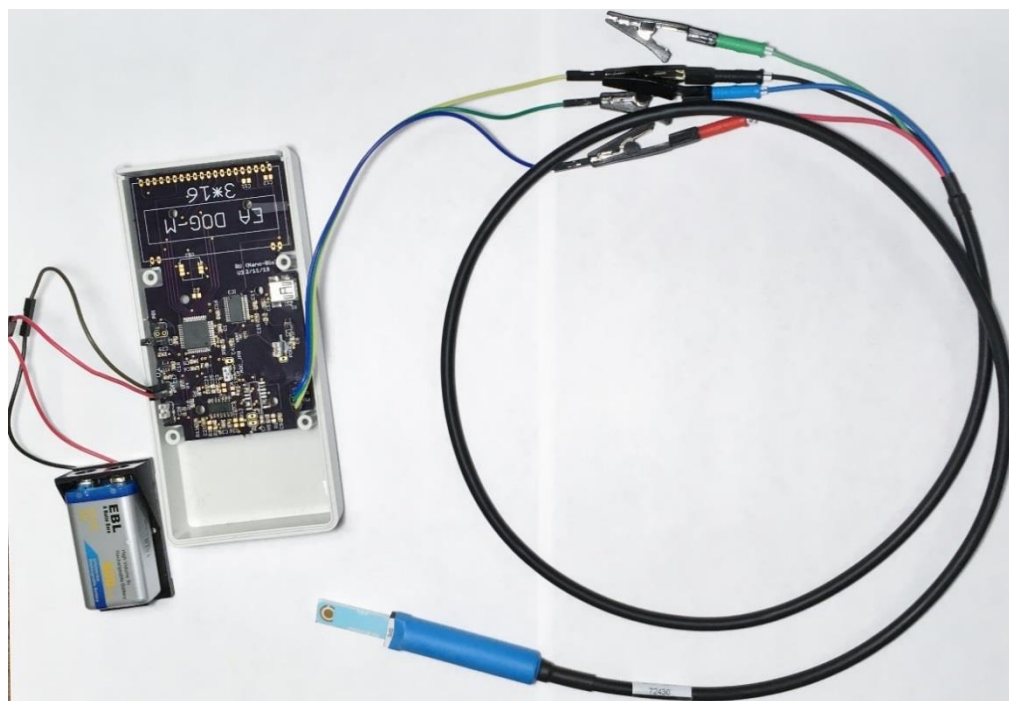


Figure 4.1 Experiment setup

We tested our circuit on Potassium ferro cyanide first to test the circuit on safer solutions first later when we were able to get distinct peaks, we moved on to lead. The lead experiment was tested on 3 different concentration; 200 ug/L, 400 ug/L and 800 ug/L of lead was mixed in solution of nitric acid/acetate buffer. A linear sweep Square-wave stripping Voltammetry was conducted on the solution. Following were the specifications designed for the experiment:

Electroplating time: 240 sec.

Electroplating Voltage: -1V

Scan rate: 125mv/sec

Initial Voltage: -1V

Final Voltage: -0.2V

Step size: 5mv

## **4.2 Result Analysis:**

The measured voltages were analyzed in MS excel. First the measured voltage was converted into current values depending on the Feedback resistor. Then the applied voltage was modified to represent the voltage with respect to the Reference electrode. Then the measured current was plotted with respect to the applied voltage. Later the buffer solution was analyzed in Origin software to create a baseline. This baseline was subtracted from the current values to measure the peak currents. The following Figure shows the results we were able to plot for the various concentrations.

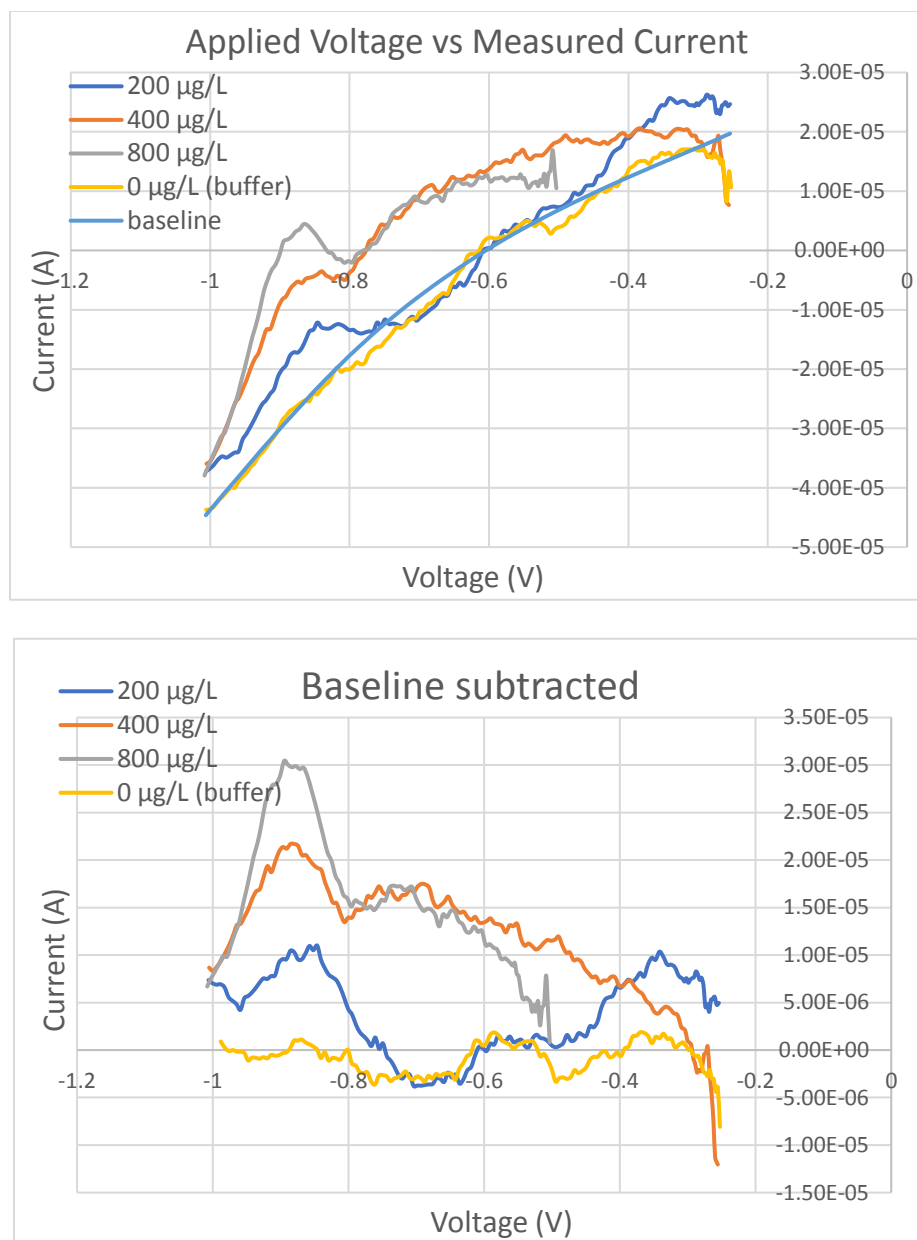


Figure 4.2 Measured Current vs Applied Voltage

The above figure shows the actual read current waveforms and the current values after baseline subtraction. In the first part of the figure, the current levels are constantly rising as a ramp because it follows the applied voltage ramp which is from -0.9V to -0.2V. The

three concentrations under consideration are the 200 $\mu\text{g/L}$ , 400 $\mu\text{g/L}$  and 800 $\mu\text{g/L}$ . The 200 $\mu\text{g/L}$  peak represents a maximum current of a 10.5 $\mu\text{A}$ , while the 400 $\mu\text{g/L}$  solution represents max current of 21.5 $\mu\text{A}$  and 800 $\mu\text{g/L}$  solution has 30 $\mu\text{A}$  max current. The yellow waveform is the current level of the buffer solution and as the buffer has no content of lead it does not have the characteristic peak. We evaluated these results using the Gamry 600+ Potentiostat. The peak currents match our experimented results. We also found that the expected current follows a linear relationship with the concentration. Thus for distinct detection of higher current some of the experiment parameters must be changed i.e reducing the scan rate or the electroplating time. Following figure shows the peak current for the different concentrations we tested at the redox potential of the solution.

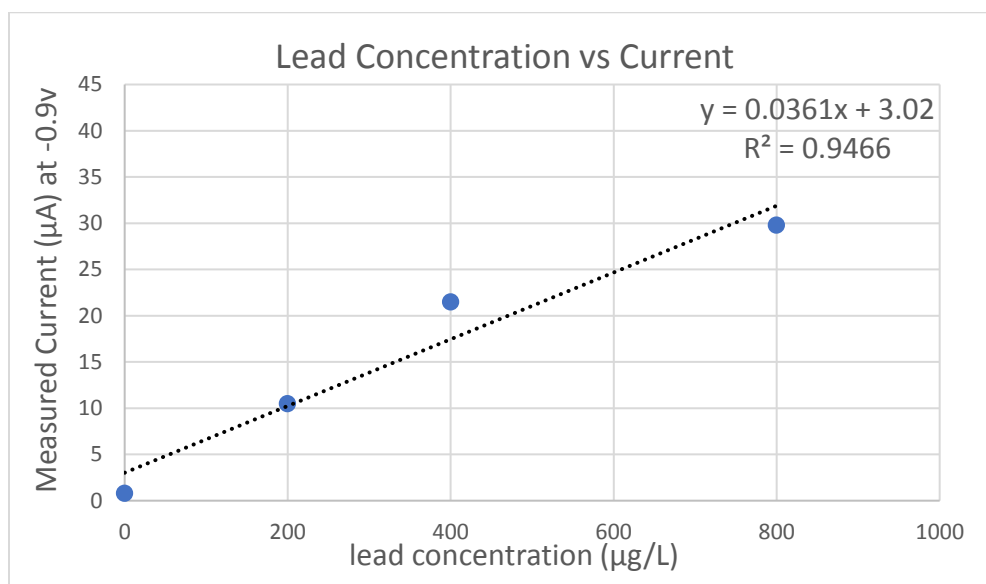


Figure 4.3 Variation of peak Current with concentration

## **Chapter 5**

### **Conclusion and Future Scope**

Thus, we were able to build and test a miniature portable precise Potentiostat for lead level detection. The experimental results show that we can measure lead levels as low as 200 ug/L. But the current levels can very well suggest we can go much lower than that. The higher lead level detection limit can go till ten thousand ug/L. We also designed a user-friendly data acquisition software program which can further be designed to have an automatic baseline subtraction and smoothening of the curve. The future scope of this project will be to create a water-resistant model of the device to make it more robust. It can also be modified to have a dedicated power source and wireless data transfer. One of the major problems we faced was that of the voltage transients at the reference electrodes, these transients were reduced by bypassing capacitors, but it causes higher current consumption, thus a better work around can be thought of.

## Chapter 6

### Appendix

#### 6.1 Sensor Fabrication:

Screen Printed Carbon Electrodes C 110 designed by Metrohm company was used for experiments. The electrodes are fabricated on a ceramic surface, first an adhesive is applied on the ceramic base, then the electrode metal plates are pasted on it and later the while electrode surface is insulated with blue plastic film. Only the ends are left uncovered, one where the circular working electrode and the annular counter electrode is, and the other end which is plugged into the SPE cable. The following figure shows the structure of the electrode [6]. The counter and the working electrode (4mm diameter) is made of carbon while the Reference electrode is made of Ag/ AgCl.



Figure 6.1 SPE Carbon electrode

## 6.2 PCB Design:

Two-layer pcb was redesigned on the Eagle CAD software, upper layer containing the required components and a ground plane covering the lower level to reduce the electrical noise and to reduce any interference. Power supply bypassing was done by adding ferrite beads to the power supplies to reduce any possible voltage spikes. Bypassing and feedback capacitors were used at opamp terminals to reduce the noise and to avoid self-oscillations [8]. Also high value capacitors were used at the reference electrodes to reduce the voltage transients during plugging the power supply. The board contains connectors to attain some flexibility to working of the circuit.

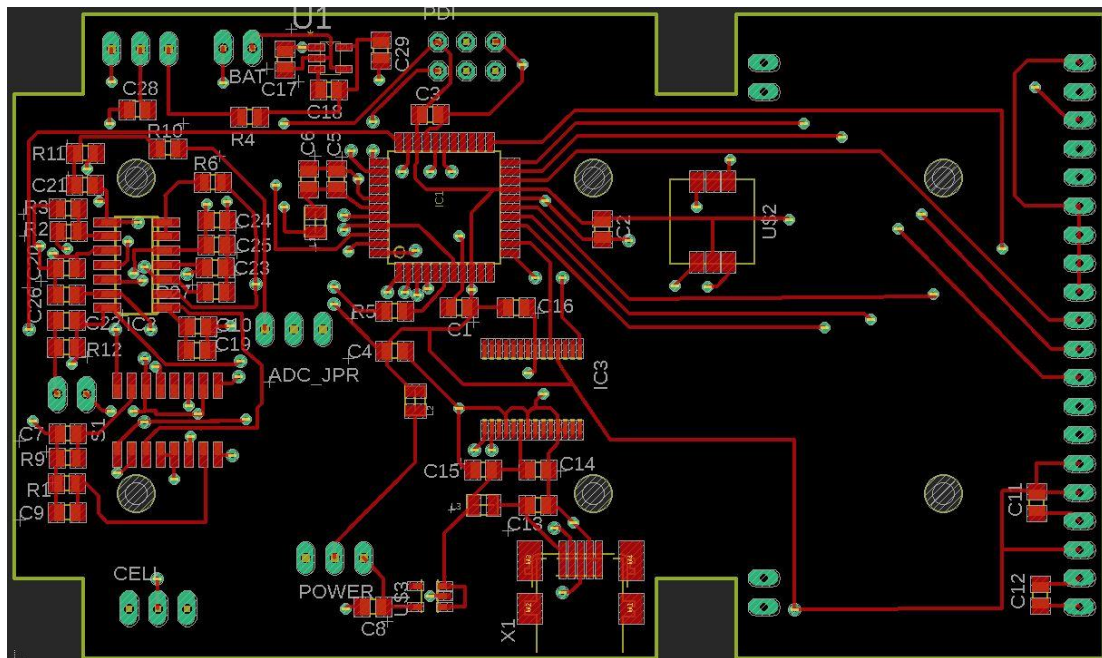


Figure 6.2 PCB layout top layer

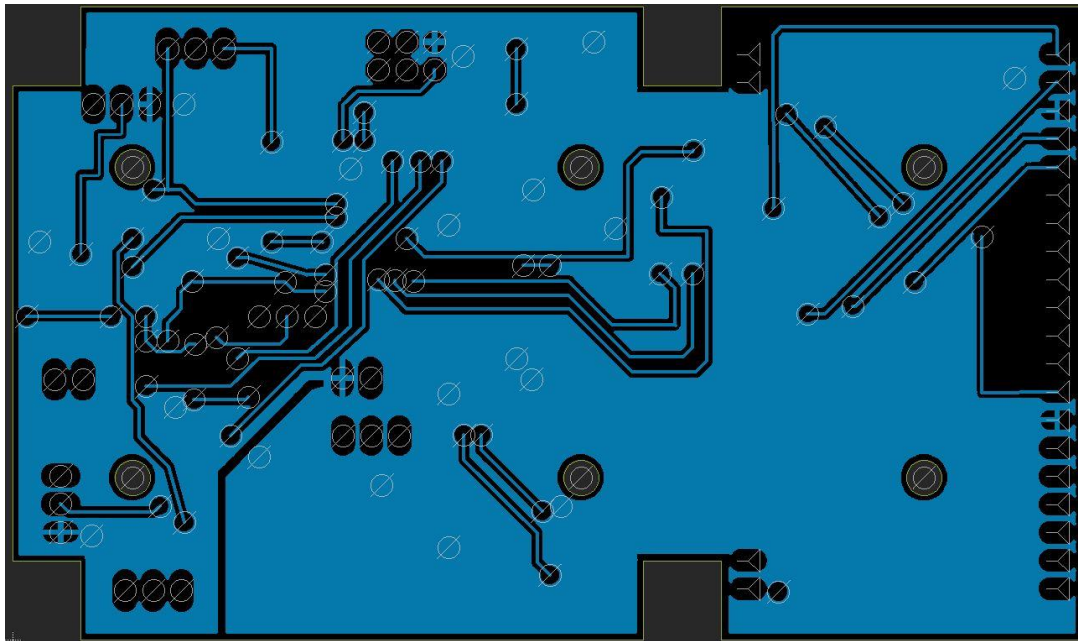


Figure 6.2 PCB layout Bottom layer

### 6.3 Atmega 32A4-AU Microcontroller

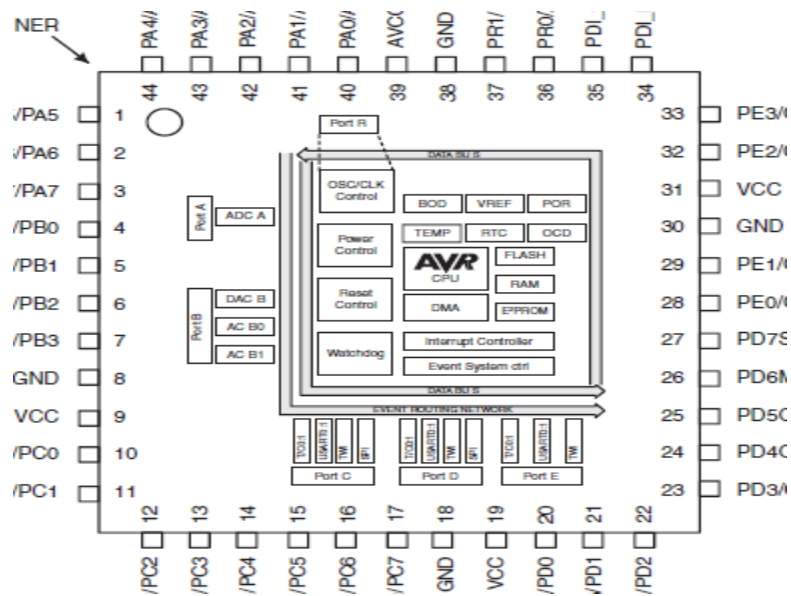


Figure 6.3 Atmega 32A4-AU block diagram

It is a 32-bit microcontroller with one 12-channel 12-bit ADC, one 2-channel 12-bit DAC and five USARTs. It has advanced programming, testing and Debugging interface with 5 GPIO ports [7]. It also has an internal oscillator and has an operating voltage of 1.8v – 3.6v. Port A was used to read the data from the experiment through an ADC and port B was used to generate the desired signal through a DAC. While port C was used for serial data transfer from the microcontroller to the computer. Port D and E can be used for connecting an LCD, flash memories etc. Atmel ICE programmer is used to program the board through “PDI clock” and “PDI data” pins.

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