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Optimizing the AD5940 for Electrochemical Measurements

by Micheál Lambe

INTRODUCTION

The AD5940 is a high precision, ultra low power, Analog Front End (AFE) system designed to excite and measure a sensors current, voltage or impedance response.

The AD5940 is specifically designed for high precision analysis of electrochemical cells. This application note details how to set up and optimize the AD5940 to perform electrochemical measurements on a typical electrochemical cell.

The hardware used is the AD5940 evaluation kit, which includes the EVAL-ADICUP3029 Arm Cortex™-M3 microcontroller-based Arduino Uno form factor, the EVAL-AD5940ARDZ evaluation board, and the AD5940Sens2 daughter card, shown in Figure 1.

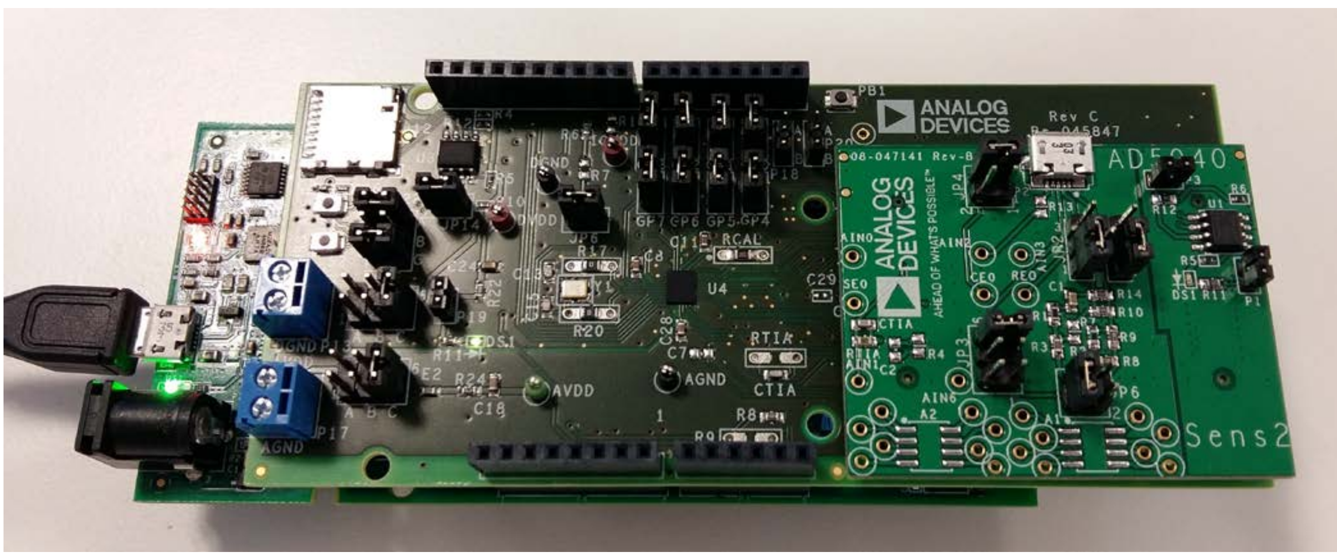

Figure 1. Hardware Setup

TABLE OF CONTENTS

REVISION HISTORY

x/2018—Revision 0: Initial Version

EVALUATION KIT

CONDITIONS REGARDING THE USE OF THIS PRODUCT IN HEALTHCARE APPLICATIONS

In addition to the terms found at the end of this document, the following shall also apply to the use of the EVAL-AD5940BIOZ evaluation kit:

This evaluation board design is being provided "as is" without any express or implied representations or warranties of any kind and the use of this board or design shall impose no legal obligation on Analog Devices, Inc., and its subsidiaries, employees, directors, officers, servants and agents. In addition, it is understood and agreed to that the evaluation board or design is not authorized for use in safety critical healthcare applications (such as life support) where malfunction or failure of a product can be expected to result in personal injury or death. This board must not be used for diagnostic purposes and must not be connected to a human being or animal.

This evaluation board is provided for evaluation and development purposes only. It is not intended for use or as part of an end product. Any use of the evaluation board or design in such applications is at your own risk and you shall fully indemnify Analog Devices, Inc., its subsidiaries, employees, directors, officers, servants and agents for all liability and expenses arising from such unauthorized usage. You are solely responsible for compliance with all legal and regulatory requirements connected to such use.

EVALUATION KIT CONTENTS

- EVAL-AD5940ARDZ evaluation board
- EVAL-ADICUP3029 evaluation board
- AD5940Sens2 daughter card
- Micro USB to USB cable

AD5940 MEASUREMENT LOOP

The AD5940 data acquisition loop consists of a low bandwidth loop, a high bandwidth loop, a high precision analog-to-digital converter (ADC), and a programmable switch matrix.

The low bandwidth loop consists of the following:

- Low power, dual output digital-to-analog converter (DAC) that generates V_{ZERO}nd V_{BIAS}.
- Low power trans-impedance amplifier (TIA) used to convert current to voltage. It ensures that the dc voltage in the sensor is zero when the dc voltage of the DAC and the TIA bias voltage are equal.

The high bandwidth loop consists of the following:

- High speed DAC and excitation amplifier designed to generate a high frequency ac excitation signal when making impedance measurements.
- High speed TIA designed to convert high bandwidth current signals up to 200 kHz into voltages to be measured by the ADC.

The switch matrix consists of the following:

- A series of programmable switches that allows connection of external pins to the high speed DAC excitation amplifier and to the high speed TIA inverting input.
- A series of programmable switches that allows calibration of unknown sensor impedance vs. an external known resistor.
- A series of programmable switches that allows connection of external TIA gain resistor.
- A series of programmable switches that allows connection of external calibration resistor (RCAL).

ELECTROCHEMICAL/POTENTIOSTAT MEASUREMENT THEORY

Electrochemical measurements are measurements carried out on an electrochemical cell. Common examples of electrochemical cells are electrochemical gas sensors, Blood Glucose strips and continuous glucose monitoring patches.

An electrochemical cell typically has three electrodes: the counter, reference and sense (sometimes known as working). Two electrode and four electrode variants also exist though less common.

In normal operation, a voltage is applied between the reference and sense electrodes. This is known as the sensor's bias voltage. Electrochemical reactions occur at the sense electrode where a current is generated and measured. A potentiostat circuit is required to maintain the sensor bias voltage and sink/source current as it is required when the electrochemical reactions take place. The current is sink/sourced through the counter electrode. Figure 2 shows a typical electrochemical cell connected to a potentiostat circuit, labelled PA.

This application note will describe how to carry out the following electrochemical measurements using the AD5940:

- Amperometric
- Chrono-amperometric (pulse test)
- Cyclic voltammetry
- Square Wave Voltammetry
- Electrochemical impedance spectroscopy (EIS)

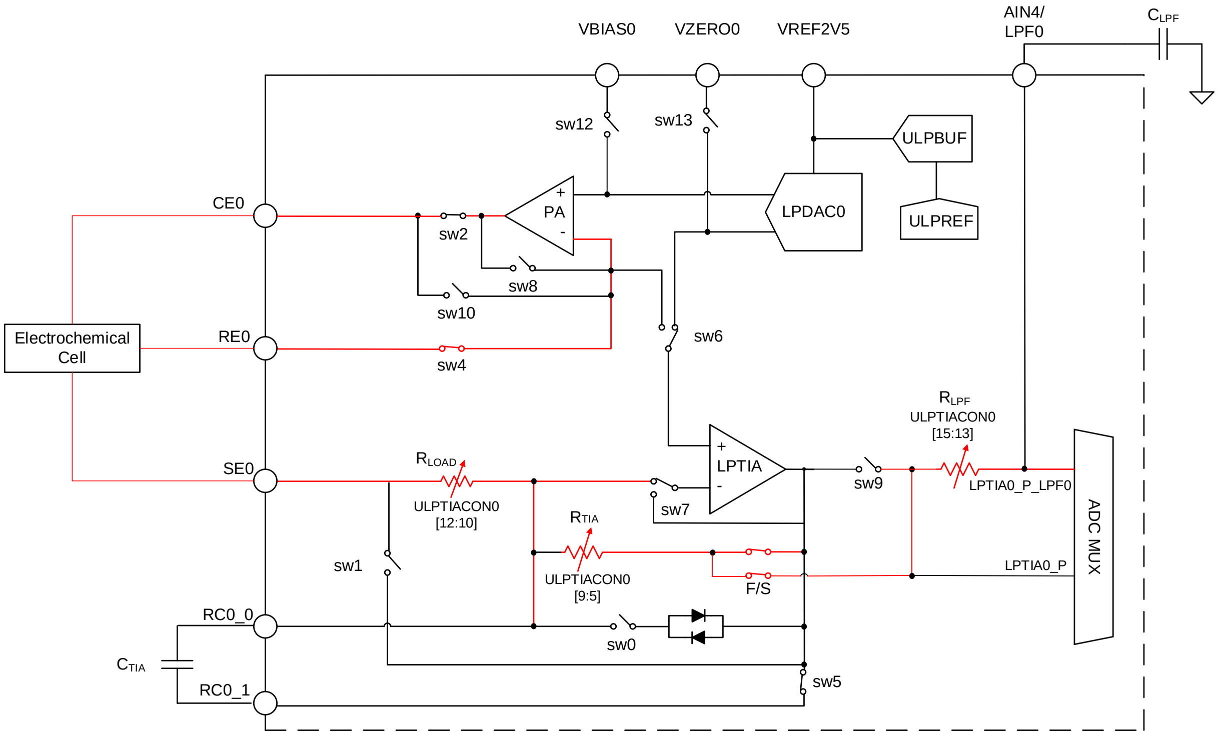

Figure 2. Electrochemical Cell Connected to AD5940

AMPEROMETRIC

Amperometric measurement is a basic electrochemical measurement where a bias voltage is applied to a sensor and the response current is monitored. The AD5940 uses its low power DAC, potentiostat amplifier, and low power TIA (LPTIA) to set a voltage on the sensor and measure the current. The low power DAC is a dual output DAC with a 12-bit option and a 6-bit option. The 12-bit output, known as V_{BIAS}, sets the voltage on the counter and reference electrode. The 6-bit output, known as V_{ZERO}, sets the voltage on the working electrode, also know as sense electrode or SE0. For zero bias sensors, the V_{BIAS} and V_{ZERO} outputs are set to the same value, which is optimally 1.1 V. For a nonzero bias sensor, the bias across the sensor is set by adjusting V_{BIAS} and V_{ZERO}.

HARDWARE CONFIGURATION

To set up the hardware, plug the EVAL-AD5940ARDZ evaluation board into the Arduino connectors on the EVAL-ADICUP3029. Plug the AD5940Sens2 daughter card into the EVAL-AD5940ARDZ evaluation board. On the AD5940Sens2 board, ensure the following jumpers are connected:

- JP1 – Jumper on PIN1 and PIN2
- JP2 – Jumper on PIN1 and PIN2
- JP3 – Jumper on PIN5 and PIN6
- JP6 – Jumper on PIN1 and PIN2

Connect the EVAL-ADICUP3029 to the PC via the micro USB cable as in **Error! Reference source not found.**.

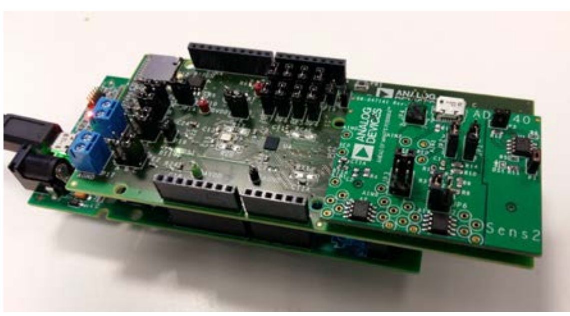

Figure 3: Evaluation board setup

MEASUREMENT EXAMPLE

The AD5940 software development kit has a dedicated amperometric measurement example.

To run amperometric measurements on the evaluation hardware, there are the following options:

- SensorPal graphic user interface (GUI) tool
- IAR Embedded Workbench® firmware example

For quick prototyping, use SensorPal. Details on installing SensorPal can be found in the Getting Started Guide {Where can this guide be found? This will be uploaded on the web. Currently with Com services UG-1292}. There are a number of configurable parameters to define measurements and SensorPal provides a quick graphing mechanism.

Alternatively, open the **AD5940_Amperometric** project in IAR Embedded Workbench.

To modify default measurement parameters, use the AD5940AMPStructInit() function. There is a data structure, **AppAMPCfg**, that contains the configurable parameters. More details on the firmware examples can be found in the doc folder.

RUNNING THE EXAMPLE

To build the project, right click on the project in the **Workspace** window and click **Rebuild All**, as shown in Figure 4.

Figure 4. Project Build

To download the code to the microcontroller and begin a debug session, click on the green play icon. Click **Download and Debug (Ctrl+D)** to start running the code (see Figure 5).

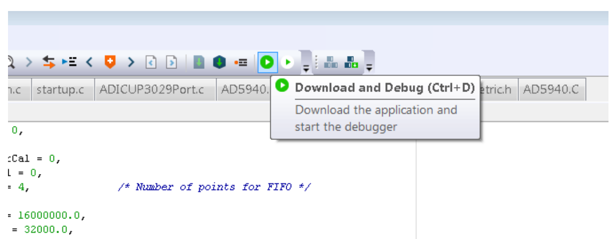

Figure 5. Starting the Debugger

To view the measurement data, open a terminal program, such as RealTerm. Set the **Baud** rate to **230400** and connect to the relevant communication (COM) **Port**.

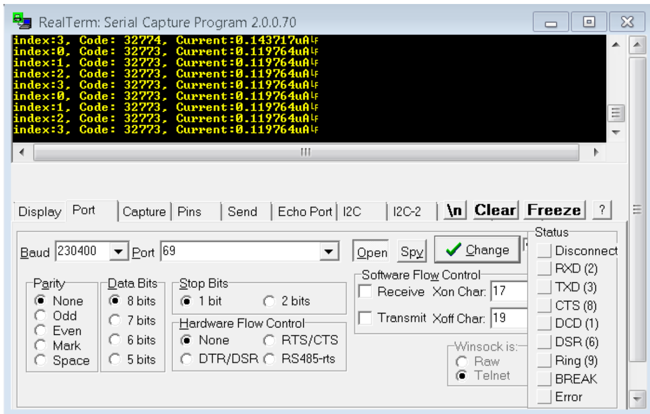

Figure 6. Data Displayed on the Terminal

If the SensorBias is set to zero, there will be no voltage between the reference and sense electrodes, thus the current displayed will be zero. Try modifying SensroBias to 300mV. A current of roughly 353µA will be measured. To verify this is correct measure the voltage between RE0 and SE0 and calculate the current using the following formula:

$I = V(RE\text{-}SE)/750\Omega$

Note, the voltage between RE0 and SE0 wont be exactly 300 mV as expected. This is because there is only a resistor connected and the voltage on RE0 and SE0 will try to match each other.

CHRONO-AMPEROMETRIC (PULSE TEST)

Chrono-amperometric, or pulse test, is a test in which the voltage across the counter electrode and sense electrode is pulsed. The current response is measured on the sense electrode through the LPTIA.

In normal operation, the AD5940 sets the voltage on the counter electrode via VBIAS and the voltage on the sense electrode is set via VZERO. These are typically set to 1.1 V for a zero bias sensor. In this scenario, applying a pulse voltage on VBIAS applies a pulse on the sensor. The response current can be measured using the low power TIA or the high speed TIA, depending on the speed of the response required.

HARDWARE CONFIGURATION

Using the same hardware configuration as in the previous section, move the jumper on JP6 from PIN1 and PIN2 to PIN3 and PIN4. This connects the RC network shown in Figure 7 to the AD5940.

MEASUREMENT EXAMPLE

At time zero, the counter electrode is held at midscale (1.1 V) and the sense electrode is held at common mode of 1.1 V to ensure that no current flows through the sensor. Then a step voltage is applied to the sensor by increasing VBIAS. The resultant current flows through the sensor and is converted to a voltage using the low power TIA.

Figure 7 shows the RC network used for the pulse test.

Note the following:

- C_S = 10 µF
- R_S = 6.8 kΩ

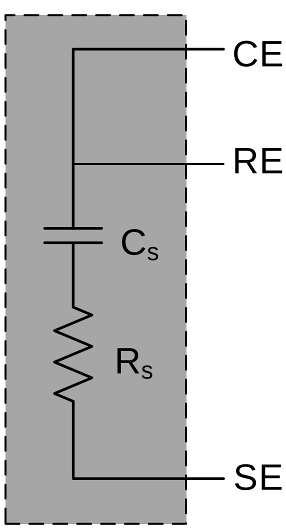

Figure 7. Sensor Configuration for Pulse Test

THEORETICAL CALCULATION

The RC time constant (τ) is the time constant of an RC circuit. It is the time required to charge or discharge the capacitor through the resistor by 63.2% of the difference between the initial and final value. For an RC circuit, $\tau = R \times C = 6.8\ k\Omega \times 10\ \mu F = \sim 68$ ms.

Peak current is measured at t1, immediately after step is applied. The capacitor is short circuit, I_{PEAK} = 500 mV / 6800 = 73.52 µA.

To calculate suitable RTIA value

$R_{TIA} = 0.9 / I_{MAX}$,

0.9 / 73.52 µA = ~12241.

10 kΩ of internal RTIA is selected to allow some head room. RTIA can have a 10% error that is calibrated out.

RUNNING THE EXAMPLE

The AD5940 software development kit has a dedicated Chrono Amperometric measurement example.

To run cyclic chrono-amperometric measurements on the evaluation hardware, there are the following options:

- SensorPal GUI tool
- IAR Embedded Workbench firmware example

For quick prototyping, use SensorPal. There are a number of configurable parameters to define the measurement and SensorPal provides a quick graphing mechanism.

Alternatively, open the **AD5940_ChronoAmperometric** project in IAR Embedded Workbench.

In the **AD5940_Main.c** file, there is an initialization function used to configure the measurement parameters (AD5940AMPStructInit()). Use this function to configure parameters such as pulse length, pulse amplitude etc.

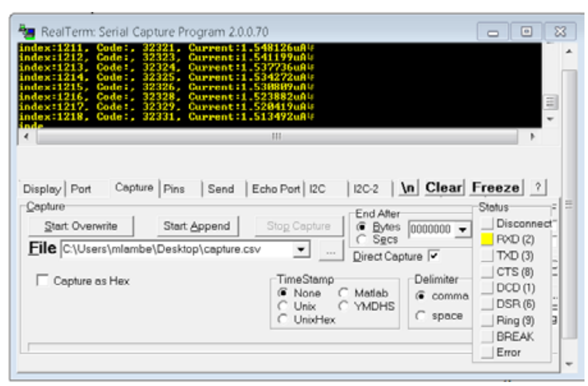

Figure 8. Terminal Program Displaying Results

To save results to a new file, click on the **Capture** tab. Select a suitable location and save the file with a **.csv** extension. Open the file with Microsoft® Excel to create a graph of the data.

The graphs below show the raw ADC code plotted against time and the current response plotted against time. Comparing the calculated theoretical peak current of 73.52µA to the peak of the transient in Figure 10 shows the measurement is correct.

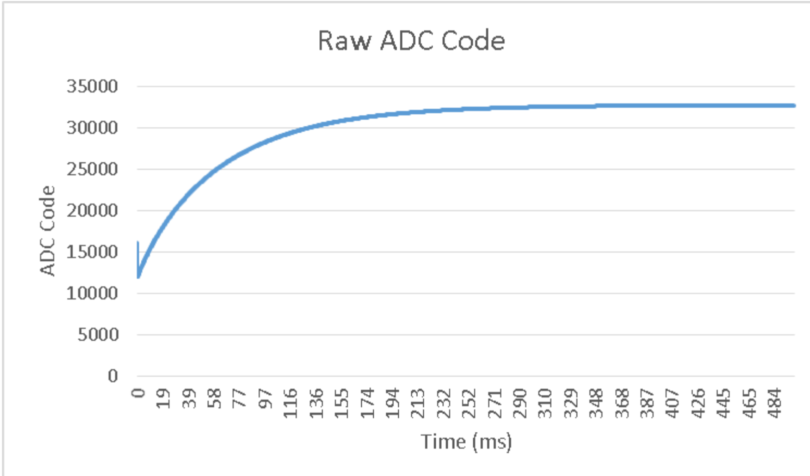

Figure 9. Raw ADC Code

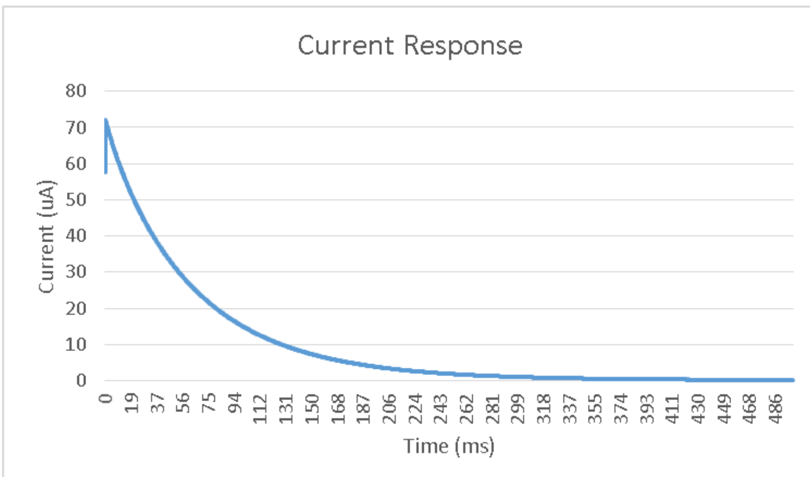

Figure 10. Current Response

CYCLIC VOLTAMMETRY

Cyclic voltammetry is an electrochemical measurement in which the voltage applied to an electrochemical cell is incremented, then decremented, linearly in a triangular shape to a point. The response current on the working electrode is measured.

To carry out this measurement on the AD5940, VZERO is set to output a voltage of 1.3 V. VBIAS can sweep from 0.3 V to 2.3 V, giving a ±1 V sweep. The response current is measured using the low power TIA.

HARDWARE CONFIGURATION

Using the same hardware configuration as in the previous section, move the jumper on JP6 from PIN3 and PIN4 to PIN1 and PIN1 as was the case for the amperometric example. This connects the 750 Ω resistor to the AD5940.

MEASUREMENT EXAMPLE

The AD5940 software development kit has a dedicated ramp measurement example.

To run cyclic voltammetry measurements on the evaluation hardware, there are the following options:

- SensorPal GUI tool
- IAR Embedded Workbench firmware example

For quick prototyping, use SensorPal. There are a number of configurable parameters to define the measurement and SensorPal provides a quick graphing mechanism.

Alternatively, open the **AD5940_Ramp** project in IAR Embedded Workbench.

In the **AD5940_Main.c** file, there is an initialization function used to configure the measurement parameters (AD5940RampStructInit()). Use this function to configure parameters such as start voltage, peak voltage, etc.

THEORETICAL CALCULATION

A suitable R_{TIA} must be selected to maximize measurement accuracy. Calculate the estimated maximum current of the sensor. For example, using the AD5940Sens2 daughter card , place a jumper on JP6 between PIN1 and PIN2. Doing this connects a 750 Ω resistor network to the AD5940. Set the RampPeakVolt to 2.3 V, giving a peak voltage of 1 V on the sensor.

I_{MAX} is calculated using the following equation:

I_{MAX} = V / R =1 / 750 = 1.33 mA. RTIA is calculated using the following equation:

$R_{TIA} = 0.9 / IM_{AX}$

$R_{TIA} = 675\ \Omega$.

Where I_{MAX} is the maximum estimated current,

R_{TIA} is calculated gain resistor.

The closest internal resistor value to 675 Ω is 200 Ω. Therefore, the internal 200 Ω R_{TIA} is selected.

RUNNING THE EXAMPLE

Rebuild the project and launch the debugger interface. Open RealTerm to capture data. Save the data with a **.csv** extension so the file can be opened in excel for analysis. Figure 11 shows a plot of response current for the ramp example.

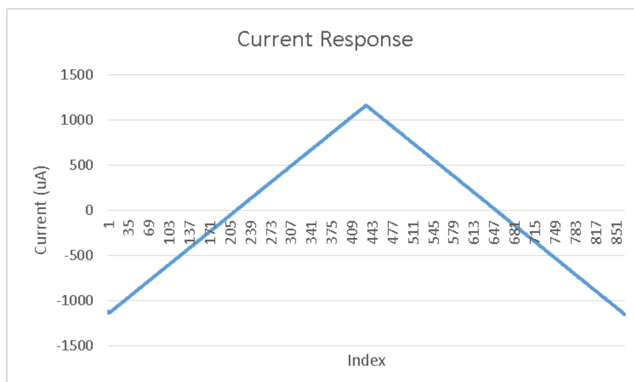

Figure 11. Ramp Current Response

SQUARE WAVE VOLTAMMETRY

Square-wave voltammetry is an electrochemical technique where the voltage between the reference and sense electrode is incremented in a square wave fashion as in Figure 12. The response current on the working electrode is measured after each half step.

This technique is similar to cyclic voltammetry method outlined in the previous section.

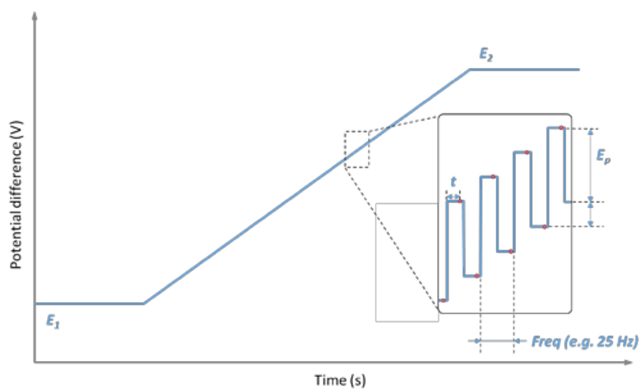

Figure 12: Square Wave Voltammetry

HARDWARE CONFIGURATION

Remains the same as per Cyclic Voltammetry example.

MEASUREMENT EXAMPLE

The AD5940 software development kit has a dedicated square wave voltammetry measurement example, AD5940_SqrWaveVoltammetry.

To run the square wave voltammetry measurements on the evaluation hardware, there are the following options:

- SensorPal GUI tool
- IAR Embedded Workbench firmware example

For quick prototyping, use SensorPal. There are a number of configurable parameters to define the measurement and SensorPal provides a quick graphing mechanism.

Alternatively, open the **AD5940_SqrWaveVoltammetry** project in IAR Embedded Workbench.

In the **AD5940_Main.c** file, there is an initialization function used to configure the measurement parameters (AD5940SWVStructInit()). Use this function to configure parameters such as start voltage, peak voltage, frequency, amplitude etc.

Table 1: Variable Mapping

Label in Diagram	**Variable name in Firmware**
E_1	SqrWvStartVolt
E_2	SqrWvPeakVolt
Freq	Frequency
E_P	SqrWvAmplitude
E_{STEP}	SqrWvStep
Delay	SampleDelay

RUNNING THE EXAMPLE

Rebuild the project and launch the debugger interface. Open RealTerm to capture data. Save the data with a **.csv** extension. Figure 13 shows a plot of response current for the SWV test incrementing the signal from -1V to 1V with E_P = 50mV and E_{STEP} = 5mV, Freq = 25Hz. Figure 14 shows a portion of the graph zoomed in to highlight the current profile.

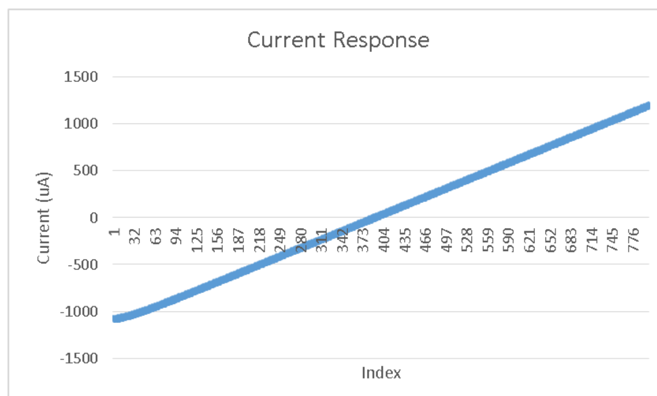

Figure 13. SWV Current Response

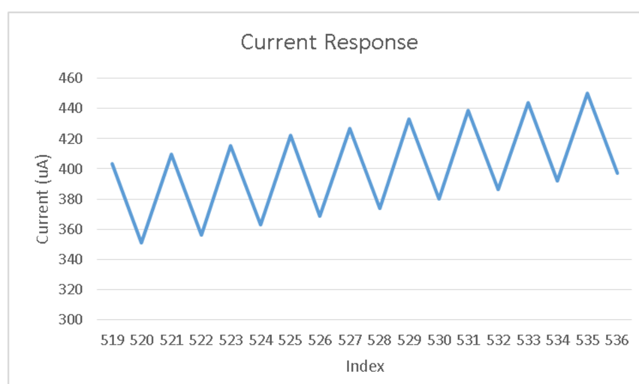

Figure 14: SWV Current Response Zoomed In

ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

EIS is an electrochemical measurement in which the impedance of the electrochemical cell is measured over a range of frequencies.

The AD5940 can carry out EIS measurements with frequencies of up to 200 kHz. Also, a dc bias applied on an electrochemical cell can be maintained even when carrying out impedance measurements.

HARDWARE CONFIGURATION

Hardware can remain the same as per previous example to measure the 750 Ω impedance. Alternatively, move the jumper on JP6 to PIN3 and PIN4 to measure the impedance of the RC network.

MEASUREMENT THEORY

The EIS measurement uses a ratio metric approach. A signal is applied across the known resistor, (RCAL) and the response current is measured. The same signal is then applied across the unknown impedance and the response current is measured. A Discrete Fourier Transform (DFT) is performed on the resulting currents to determine the magnitude and phase values of each. The unknown impedance magnitude can then be calculated using the following equation:

$$|Z_{UNKNOWN}| = \frac{I_{Rcal}Mag}{I_{Zunknown}Mag} \times R_{CAL}$$

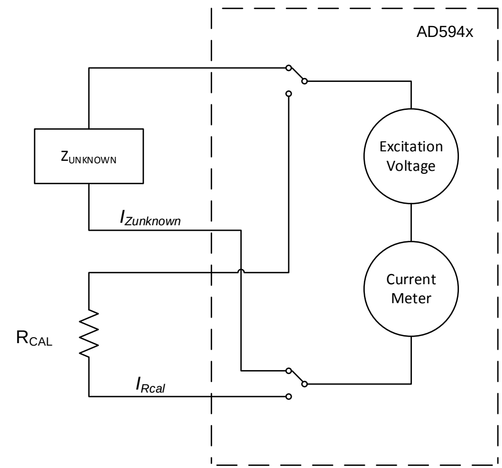

Figure 15. 2-Wire Measurement Overview

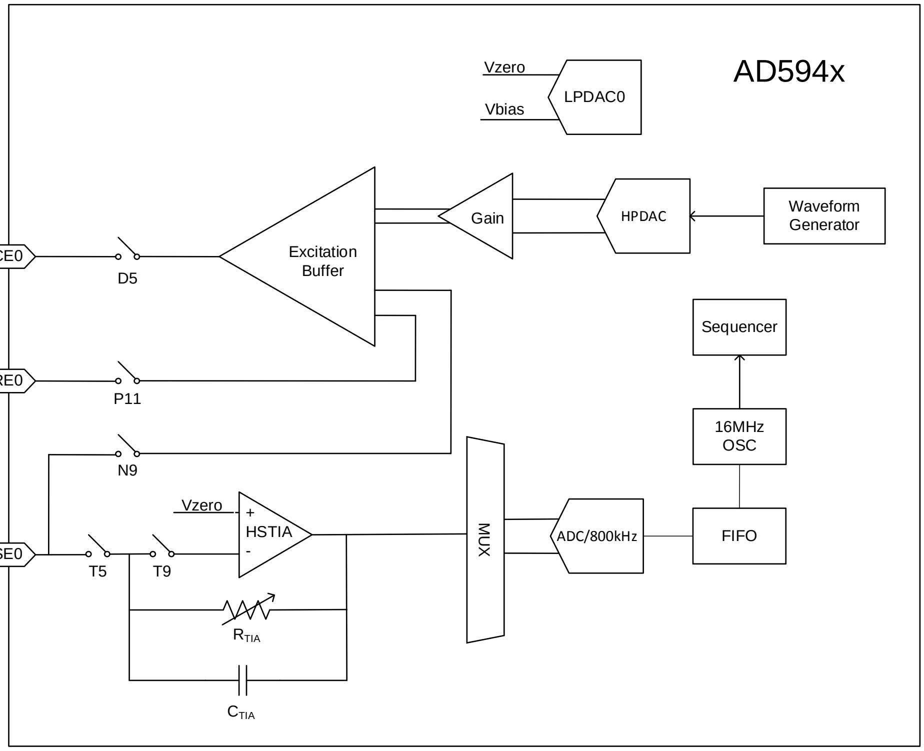

Figure 16. EIS Block Diagram

For EIS measurements, the sensor is disconnected from the low power potentiostat and LPTIA circuitry. The sensor is then connected to the High Speed DAC (HSDAC) excitation amplifier and to the High Speed TIA (HSTIA) via the switch matrix, as shown in Figure 16. The HSDAC and HSTIA are needed for high bandwidth signals where the excitation frequency is greater than 200 Hz.

MEASUREMENT EXAMPLE

The AD5940 software development kit has a dedicated EIS measurement example.

To run EIS measurements on the evaluation hardware, there are the following options:

- SensorPal GUI tool
- IAR Embedded Workbench firmware example

For quick prototyping, use SensorPal. There are a number of configurable parameters to define the measurement and SensorPal provides a quick graphing mechanism.

Alternatively, open the **AD5940_Impedance** project in IAR Embedded Workbench.

In the **AD5940_Main.c** file there is an initialisation function used to configure the measurement parameters (AD5940ImpedanceStructInit()). Use this function to configure excitation signal frequency and more. It is also possible to do a frequency sweep, which is configured in the **Impedance.c** file.

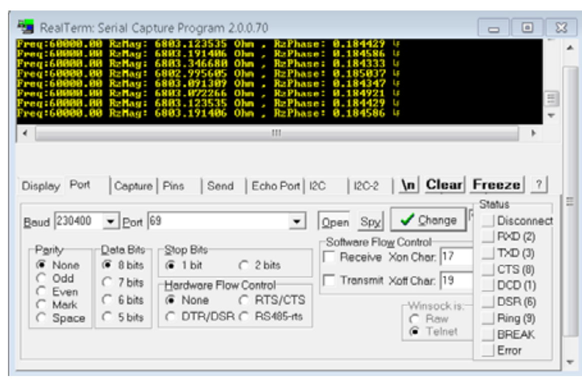

Figure 17. Impedance Results on Terminal