

# Portable Data Acquisition System for Nano and Ultra-Micro Scale Electrochemical Sensors

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**Abstract**— This work describes a flexible and portable data acquisition system that has been developed to interface to nano and ultra-micro scale electrochemical sensors at the point of use. It can perform a range of voltammetric tests, including Cyclic Voltammetry, Square Wave Voltammetry and Generator Collector Voltammetry. The data acquisition system interfaces to a smartphone, operates from a rechargeable battery and is of suitable form factor to ensure that it’s fully portable. By utilising commercially available components, this system has been developed to lower the barrier for entry for the development of emerging portable electrochemical sensing technologies at micro and nano scale. To show the full range of functionality of the system, a use case involving river water quality monitoring is presented through generation of a calibration curve, using a recently developed Tyndall National Institute ultra-microband electrochemical sensor, for the detection of dissolved oxygen in river water.

**Keywords**—Electrochemical Sensors, Portable, Data Acquisition System, App, Dissolved Oxygen Sensor

## I. INTRODUCTION

Electrochemical sensors are gaining traction for a variety of new applications including point of care clinical diagnostics and in the field environmental monitoring. To address this growing need, nano and ultra-micro scale electrochemical sensors are currently being developed by several research groups worldwide for point of sample tests [1-4]. Although many new electrochemical sensing technologies have and are being developed, it is difficult to deploy them outside of a research laboratory due to the expensive bulky benchtop equipment required to interrogate these electrochemical sensors. Currently, to achieve proof of concept at the point of measurement, an electronic engineer must be hired to create a customised solution for the sensor in question [5-7], however, even then such systems often are not sufficiently robust to deal with sensor to sensor variations that regularly occur early in the development cycle. For electrochemical sensors utilising two Working Electrodes (WE), the development of a custom acquisition system is even more onerous. While there are some commercial

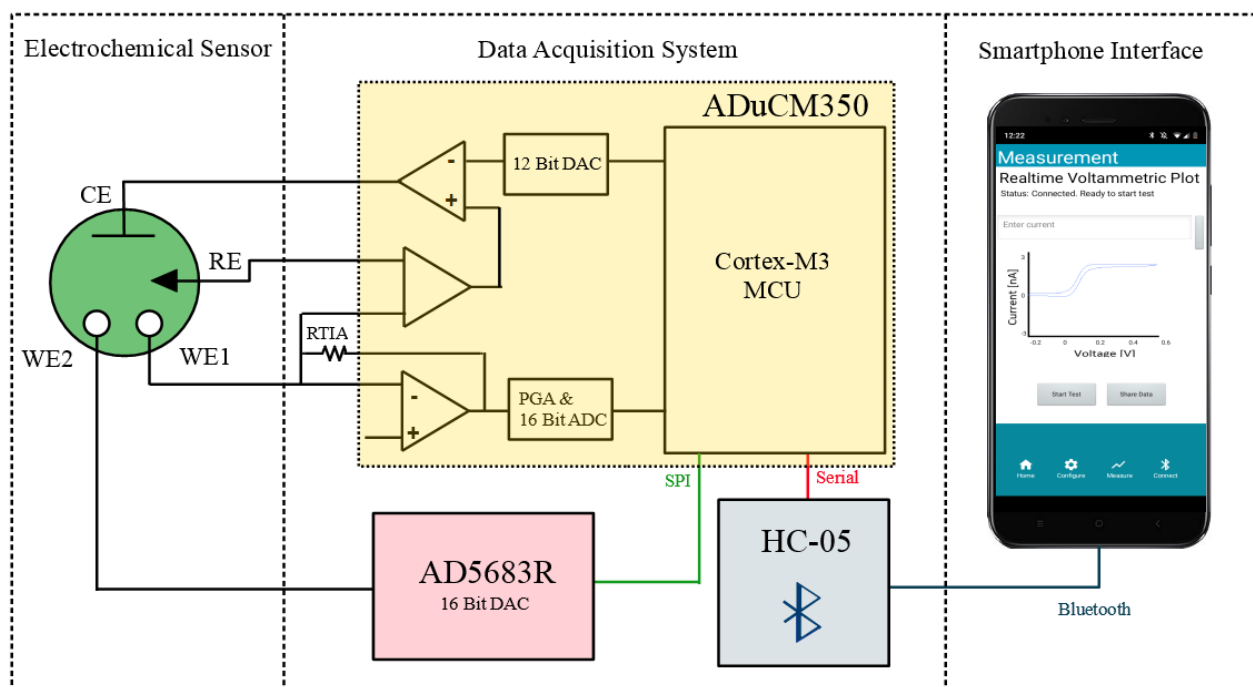


Fig. 1. Block diagram of portable point of sample system, with separate electrochemical sensor, data acquisition system and smartphone interface via Bluetooth.



Fig. 2. Front and back of data acquisition PCB, dimensions; 57 mm by 36 mm.

systems currently available [8, 9], to date they are often prohibitively expensive.

While low-cost potentiostats have been developed [10-12], they typically do not support bipotentiostatic capabilities or have the sufficient resolution to support the pico-Amp currents associated with nano and ultra-micro scale electrochemical sensors. The use of nano and ultra-micro scale electrochemical is driven by the relationship between physical dimensions of the sensor and the analyte that is being detected [13]. This work proposes a portable electrochemical acquisition system, which utilises commercial components and provides ease of use through interaction via a smartphone application and Bluetooth connection. The system can act as a low cost solution for electrochemists who want to explore their sensors capabilities in a portable or end users setting, without requiring a significant investment. It is envisaged that the system will be of particular use to nano-scale electrochemical sensing technologies that typically require the use of two WEs, which is not readily supported today.

## II. DEVELOPMENT OF SYSTEM

A block diagram of the proposed data acquisition system is presented in Fig. 1, interfacing to a two WE electrochemical sensor, and communicating back to a smartphone via Bluetooth. To enable the development of this low cost device suitable for measurements at the point of sample, a System on Chip (SoC), ADuCM350 [14], is used instead of creating a potentiostat with individual discrete components. The ADuCM350 SoC from Analog Devices is used which has an integrated potentiostatic circuit and ARM microprocessor. In addition an AD5683R Digital to Analog Converter (DAC) [15] is used to add the capability of simultaneously stimulating a second WE. A proof of concept, evaluation board based prototype of the system is described in [16]. To be fit for purpose, the system has to perform Cyclic Voltammetry (CV) and Square Wave Voltammetry (SWV) [17] while also having the additional capability of maintaining a constant potential at a second working electrode during these tests, which is known as Generator Collector Voltammetry [18].

TABLE I: SYSTEM SPECIFICATIONS

<u>General Specifications</u>			
Current Resolution	3.6pA		
Current Range	± 100nA		
Max Scan Rate	0.2 V/s		
Measurement WE Virtual Ground	1.1V		
<u>System Voltage Ranges</u>			
	WE2 (Constant Potential )		
	-0.8V	0V	0.8V
<i>Standard Mode</i>			
Voltammetric Test Range (WE1)	-0.8V to 0.3V	-0.8V to 0.8V	-0.6V to 0.8V
<i>Extended Voltage Mode</i>			
Voltammetric Test Range (WE1)	-1.9V to 1.6V	-1.1V to 1.4V	-0.3V to 2.2V

As illustrated in Fig. 1, the ADuCM350 is used to drive and control the CE (Counter Electrode) , RE (Reference Electrode) and WE 1 (Working Electrode 1). The ADuCM350 also indirectly controls the potential of the solution in the sensor that is being interrogated, through the CE using the feedback from the RE. The resultant current of the electrochemical measurement is recorded at the WE1 electrode using the integrated trans-impedance amplifier in the ADuCM350. A large external gain resistor (RTIA) of 10 M $\Omega$  is required to capture the current at a range of  $\pm 100\text{ nA}$ , which is suitable for nano and ultra-micro scale sensors [13]. For dual WE measurements the AD5683R DAC provides an additional independent stimulus.

### A. Standard Voltage Mode

The system can perform a voltammetric test in a range of  $\pm 0.8\text{ V}$  at WE1 with respect to the solution, which is sufficient for the majority of applications. This is achieved in the case of a single WE based sensor by maintaining the WE at a virtual ground (1.1V) and driving the potential of the solution, via the CE, such that the potential difference between WE1 and the solution is the value that the user specifies with respect to the RE. This varies from sensor to sensor and is also a function of the analyte under observation. As seen in Fig. 3, for dual

TABLE II: SYSTEM COST OVERVIEW

<u>Item</u>	<u>Cost (€)</u>
<b><u>PCB</u></b>	
<b>Unit</b>	8.02
<b>Components</b>	39.50
<b>Assembly</b>	37.00
	84.52
<b><u>External Components</u></b>	
<b>HC-05</b>	2.78
<b>Li Battery &amp; Charger</b>	3.00
	5.78
<b>Total</b>	90.30

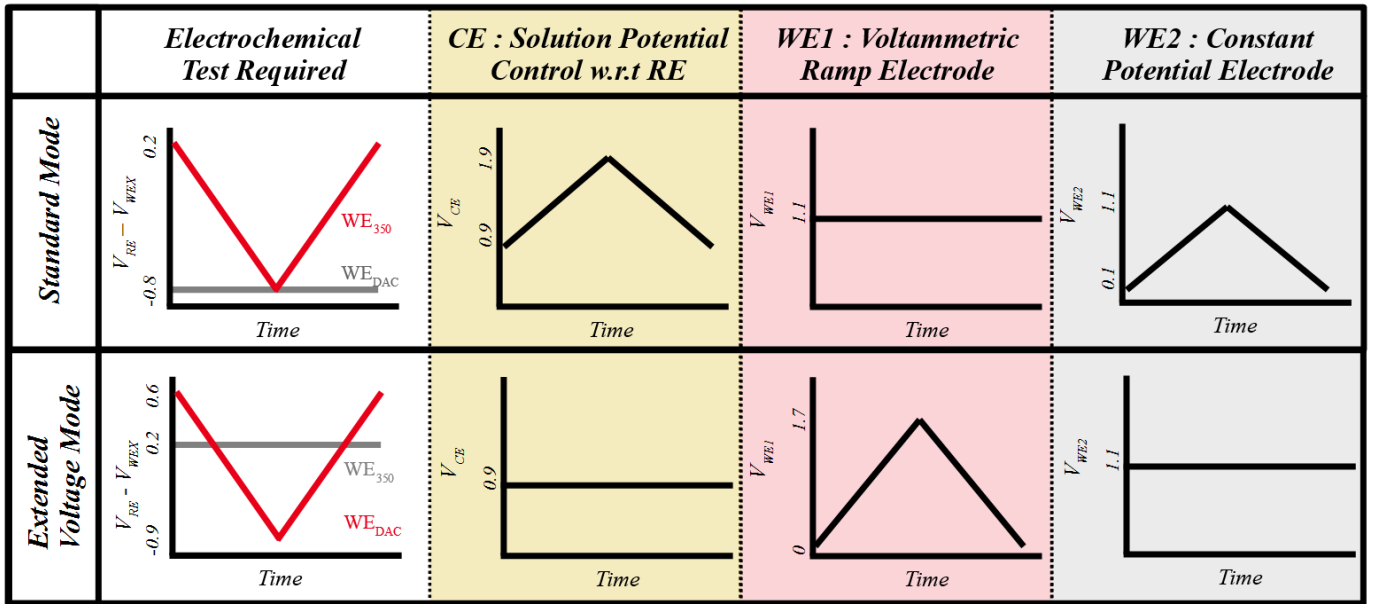


Fig. 3. Voltage levels applied by system to realise electrochemical tests required.

electrode measurements, the AD5683R is used to maintain a constant potential difference, specified by the user, relative to the CE. As depicted in Table I, the voltammogram in this mode is limited to  $\pm 0.8V$  as the DAC driving the CE in the ADuCM350 experiences headroom issues outside an absolute range of  $0.3V$  to  $1.9V$ . As seen in Table I, the range of all voltammograms for dual electrode sensing varies depending on the potential specified at WE2 (Working Electrode 2). In the standard operating mode, this is limited only by the ability of the AD5683R to mirror the potential at the CE at the specified offset. In Fig. 3, it can be seen that the proposed system is capable of supporting the requirements of this application.

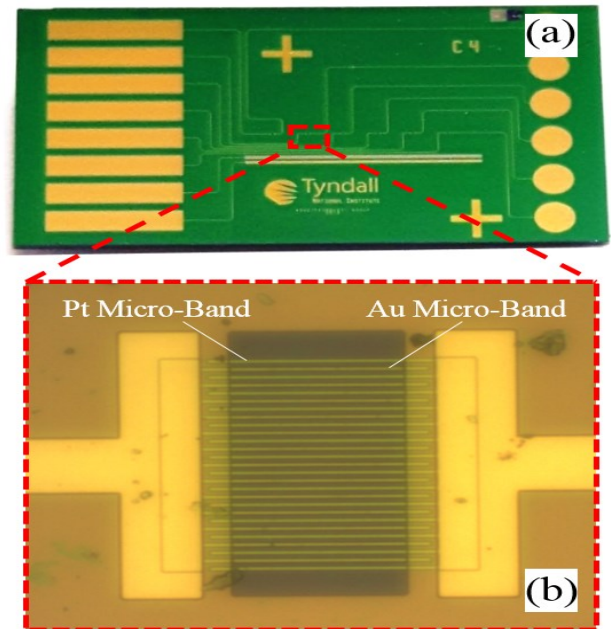
#### B. Extended Voltage Mode

In certain measurements, it is required to measure the resultant current at the working electrode, WE2, which is held at a constant potential during a dual electrode electrochemical test. The data acquisition system has been developed to integrate this capability accommodating such sensors. In the extended voltage mode, the voltage at the interface of WE2 and the solution is maintained at a constant potential through the ADuCM350, and the AD5683R is utilised to perform the necessary voltage sweep at the WE1, which is depicted in Fig. 3. In this mode there is also the possibility of applying a wider range of voltage potentials during the voltammogram as the solution is maintained at a constant potential. This allows for the full  $0V$  to  $2.5V$  range of the AD5683R DAC to be utilised, which is reflected in the wider voltammogram range seen in Table I.

Individual discrete analog components are placed as close as possible together on the PCB to limit noise, which is seen in the proximity of components on the top of each side of the PCB illustrated in Fig. 2. Due to the sensitivity of the electrochemical measurements, PCB varnish is used to insulate any exposed

vias as dirt or moisture contaminating the analog circuitry could create a constant gain error or short the potentiostatic circuit, when the system is deployed in the field. A phoenix connector enables user flexibility to interface their sensor with the system in whichever way is suitable for their sensing technology.

To allow for ease of use at the point of sample the system had to be wireless. A low-cost Bluetooth module, the HC-05 [19] is used for this purpose. An android application has been



(a) Tyndall National Institute Electrochemical Sensor chip,  
(b) Microscopic view of gold and platinum deposited  $1\mu m$  bands.  
Fig. 4. Tyndall National Institute electrochemical dissolved oxygen sensor.

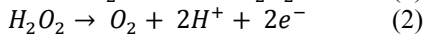
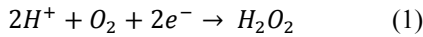
developed using MIT app inventor [20], which is a scratch based language for rapid prototyping of apps. The app allows the user to configure the necessary electrochemical test and read out the results on a graph in real-time. To ensure the adoption of the system by electrochemists, the app's user interface has been designed such that it is consistent with the interface for existing standard potentiostats. The device can be programmed to perform a range of electrochemical tests in either forward or reverse notation. A cleaning scan option is available to apply a negative potential to the working electrodes for 30 seconds prior to the test. These are essential capabilities especially when developing new electrochemical sensors. There is also a user option to save voltammetric results to a file storage service such as google drive.

The total cost of the system developed is €90.30 and a brief breakdown of costings is presented in Table. II. This is several orders of magnitude cheaper than laboratory grade equipment that have the capability of performing measurements in the nano amp range. Such a low cost should significantly lower the barrier of entry for future nano to ultra-micro scale electrochemical sensors being developed for use at the point of sample.

### III. ELECTROCHEMICAL OXYGEN SENSOR

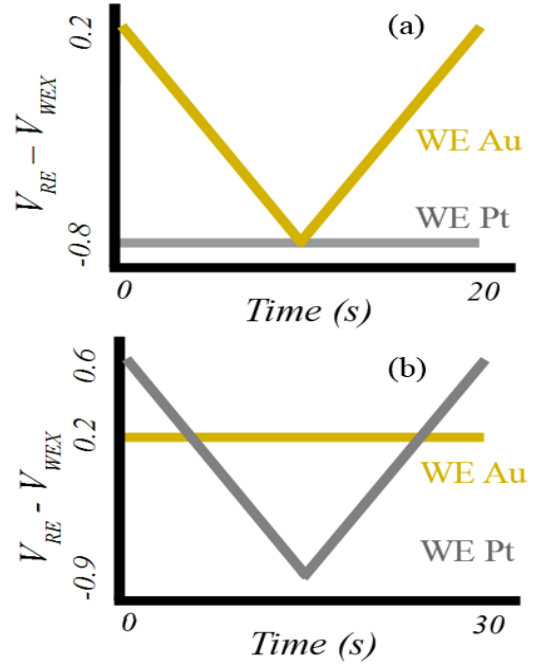
To present the full range of functionality of the system and verify its use as a portable potentiostat, a calibration curve was produced for the detection of dissolved oxygen in river water. This was performed using recently developed ultra microband electrochemical sensors [21, 22]. As seen in Fig. 4 (a), the sensor has the same topography as a Micro SD card, utilises six individual sensors, with a common integrated on chip platinum pseudo reference electrode and gold counter electrode.

The dissolved oxygen sensor consists of interdigitated platinum (WE Pt) and gold working electrodes (WE Au) microband array, shown in Fig. 4 (b). The sensor generates peroxide ( $H_2O_2$ ) at WE Au, represented by equation (1), when a potential voltage is applied. Hydrogen peroxide produced is directly proportional to the concentration of oxygen present in the sample. The generated peroxide is subsequently oxidised at WE Pt array, seen in equation (2). The electrons gained by the WE Pt in this reaction are measured and used as an indication of the oxygen level present in the sample.



A CV scan was performed on a gold electrode, to produce the peroxide while a constant potential was maintained concurrently at a platinum electrode, to re-oxidise the peroxide back to oxygen. Due to the proximity of the gold and platinum electrodes the same chemical reaction could occur a multitude of times with an individual oxygen molecule, this mechanism is known as redox cycling [21]. The redox cycling between WE Au and WE Pt electrodes increases sensitivity and selectivity of the oxygen measurement.

To execute the test using the proposed system, a CV was performed at WE Pt from 0.6 V to -0.9 V with a scan rate of



(a) Cleaning scan performed on micro-bands between dissolved oxygen measurements, (b) Voltammetric test required for dissolved oxygen measurement. Fig. 5. Voltages applied by system for dissolved oxygen measurement and electrode clean.

100mV/s, while the WE Au was maintained at 0.2 V potential throughout the test. The voltage levels applied were with respect to the platinum on chip pseudo reference electrode. The peak current of the resultant voltammogram at WE Pt corresponds to the oxygen concentration within the sample. Prior to the three tests at each concentration level, a cleaning scan was performed to remove any adsorbed residual species or platinum oxides, particularly from the platinum measurement electrode. As seen in Fig. 5 (a), to perform the cleaning scan, a

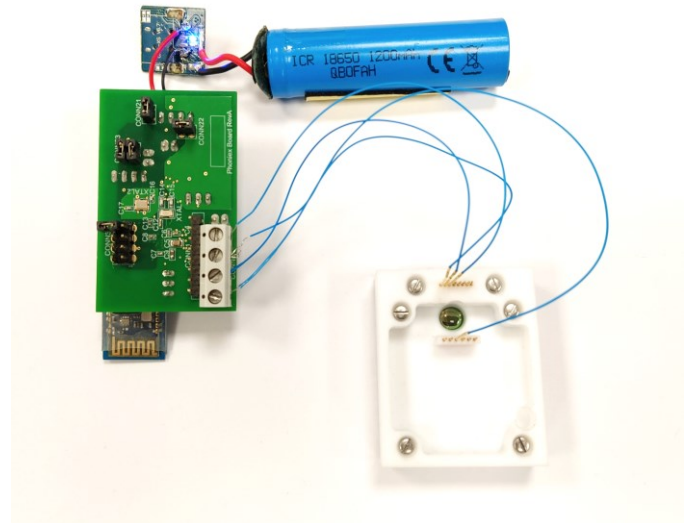


Fig. 6. Dissolved oxygen measurement apparatus.



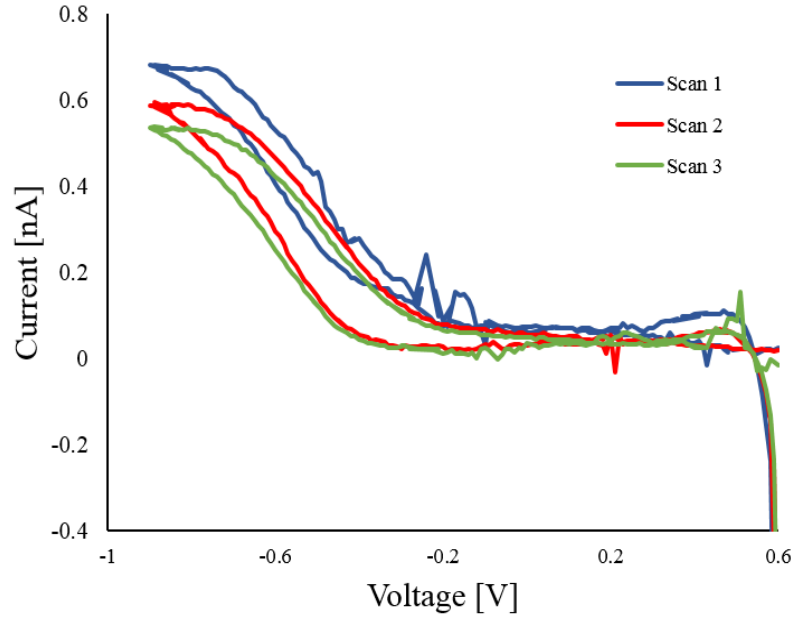
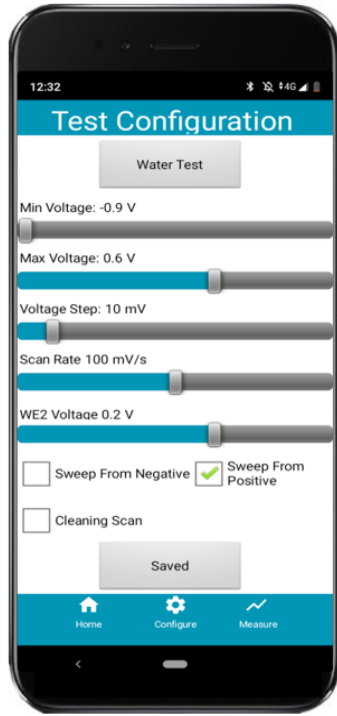


Fig. 7. Screenshot of required dissolved oxygen measurement voltammogram configuration in Android smartphone application and three resultant current outputs recorded at a dissolved oxygen concentration of 3ppm, using the developed system.

CV was performed on WE Au from 0.2 V to -0.8 V and WE Pt was maintained at -0.8 V. A scan rate of 100mV/s was used during this test.

#### IV. RESULTS

Water samples were taken from the river Lee estuary, Cork, Ireland. These samples were deoxygenated using nitrogen gas to eight different oxygen levels and a commercial dissolved oxygen probe [23] was used to confirm their concentrations. The electrochemical sensor used consisted of two interdigitated arrays of 1 $\mu$ m bands. There was an array of thirteen platinum bands and a gold band on each side of the platinum, amounting to fourteen fingers, which is depicted after use in river water under microscope in Fig. 4 (b). Measurements were taken using the proposed system three times at each concentration level in order to discern a margin of error at each point in the dissolved oxygen calibration. Due to the re-oxygenation of the sample in the sensor holder, a new water sample was used for each measurement at every concentration level.

As seen in Fig. 7, there was a clear peak for the three scans corresponding to oxygen levels in the sample. Due to the nature of the sensor and holder used in Fig. 6, long wires had to be used in the apparatus which may have led to the increased noise seen in Scan 1. Despite the imperfect apparatus setup, from a noise perspective, the results in Fig. 7 show that the proposed system is capable of performing repeatable electrochemical tests below the nano-amp current range.

Use of an anaerobic environment for the experiment was impractical due to the large number of measurements the calibration required. As a result, one of the prominent sources of error in this experiment was as a consequence of the rapid re-oxygenation of the samples in the sensor sample well as the sample is exposed to the ambient environment. This error was more prolific at reduced oxygen concentration levels, as depicted by the larger error bars at lower concentrations in the calibration plot illustrated in Fig. 8. Although the electrochemical test was initiated as soon as the sample was placed in the holder, oxygen would dissolve into the sample in transit to the sensor holder and throughout the duration of the test. The oxygen probe had an error associated with it of  $\pm$

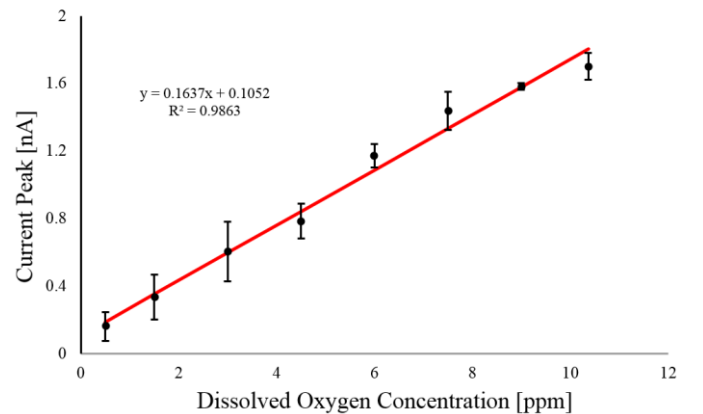


Fig. 8. Dissolved oxygen calibration plot using river water.

0.2ppm for concentrations greater than 8ppm. Although river water was also a hostile matrix for detection of the dissolved oxygen species, Fig. 8, shows that the proposed system is capable of inferring calibration curves from nano to ultra- micro scale electrochemical sensors. The resultant  $R^2$  value of 0.986 is in close alignment with results from similar sensors [20], using a laboratory grade potentiostat.

## V. CONCLUSION

In this paper, a low cost portable electrochemical data acquisition system is presented that can interface to nano and ultra-micro scale electrochemical sensors. It is low cost due to the use of widely available commercial components. Portability is realised from the small form factor and battery operation, in addition to the wireless interface via Bluetooth to a standard smartphone. The custom smartphone app, ensures simple, consistent and robust interface. The complete system has the ability to perform voltammetric tests such as CV, SWV and generator collector voltammetry. Experimental data verifies that the potentiostatic system is fit for purpose and can be used to derive calibration curves from nano and ultra-micro scale electrochemical sensors. As a result, the proposed device can be utilised as an integral tool for developing proof of concepts for emerging nano scale sensors systems at the point of test.

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