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Building a Microcontroller Based Potentiostat: A Inexpensive and Versatile Platform for Teaching Electrochemistry and Instrumentation

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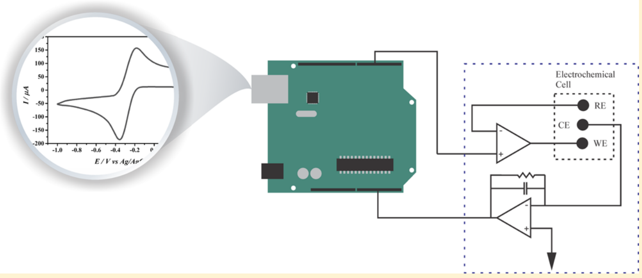
Supporting Information

ABSTRACT: A versatile potentiostat based on inexpensive and "off the shelf" components is reported. The platform was shown to be capable of performing simple electrochemistry experiments, suitable for undergraduate level teaching. The simple design and construction enable easy customization to accommodate a broad array of experimental designs. The equipment was used to calculate the diffusion coefficient of potassium ferricyanide in an aqueous solution, and the obtained result was in good agreement with the literature. Although simple in design, the low cost and good performance of the device make it a competitive alternative for teaching laboratories in the fields of both electronics and electrochemistry, and for developing teaching centers that cannot afford a commercial device.



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INTRODUCTION

Modern equipment construction and design often rely on a "black box" construction philosophy, pushing the user away from the principles of operation of the machine and transforming itself into a "data spitting" equipment.[1] Most of these design decisions are under the pretext of making the operation easy by means of a maintenance free and user-friendly equipment, but in fact, they end up preventing the full usage of the equipment capabilities and true understanding of the collected data.

With the rise of the do-it-yourself (DIY) culture and the constant growth of DIY enthusiasts, powerful prototyping platforms have become affordable and accessible for everyone. In this scope, Arduino microcontroller boards based on the ATMeaga microcontroller family stand out because of their outstanding capabilities, affordable price, and support community. Owing to these characteristics, Arduino based measuring equipment and sensors such as thermometers, pH-meters,[2] photometers,[3] and PCR thermal cyclers[4] are making their way into teaching laboratories as powerful teaching tools.[4,5] Although a few potentiostats have been reported elsewhere,[6,7] they usually rely on expensive electronic hardware and custom software in order to achieve high precision levels. On this note, we present the design and fabrication of a simple, cheap, and customizable Arduino based potentiostat that relies only on open software programs and a handful of "off the shelf" electronic components capable of performing basic electrochemical measurements in teaching laboratories. The proposed platform can be used as a multidisciplinary experiment for exploring both the electrochemistry and electronic aspects.

POTENTIOSTAT FABRICATION

Potentiostats are simple devices that rely on operational amplifiers to keep a desired potential difference between two electrodes (working and reference electrodes) immersed in a solution while recording the electrical current that flows between them. Normally a third electrode (counter electrode) is added to the system in order to isolate the electrode used as a potential reference (reference electrode) from the charge transfer reaction.[8] A basic potentiostat can be assembled using a handful of simple electronic components consisting of some resistors, capacitors, and operational amplifiers. A circuit diagram of the electronic components used in the fabrication of the proposed potentiostat and a device fabricated using a custom etched printed circuit board can be seen in Figure 1. This diagram represents the potential controlling and current measurement parts of the equipment, where all the analog electronics are. A photo of the actual device used on the measurements herein presented, fabricated using a breadboard type construction, can be seen in the Supporting Information of this manuscript.

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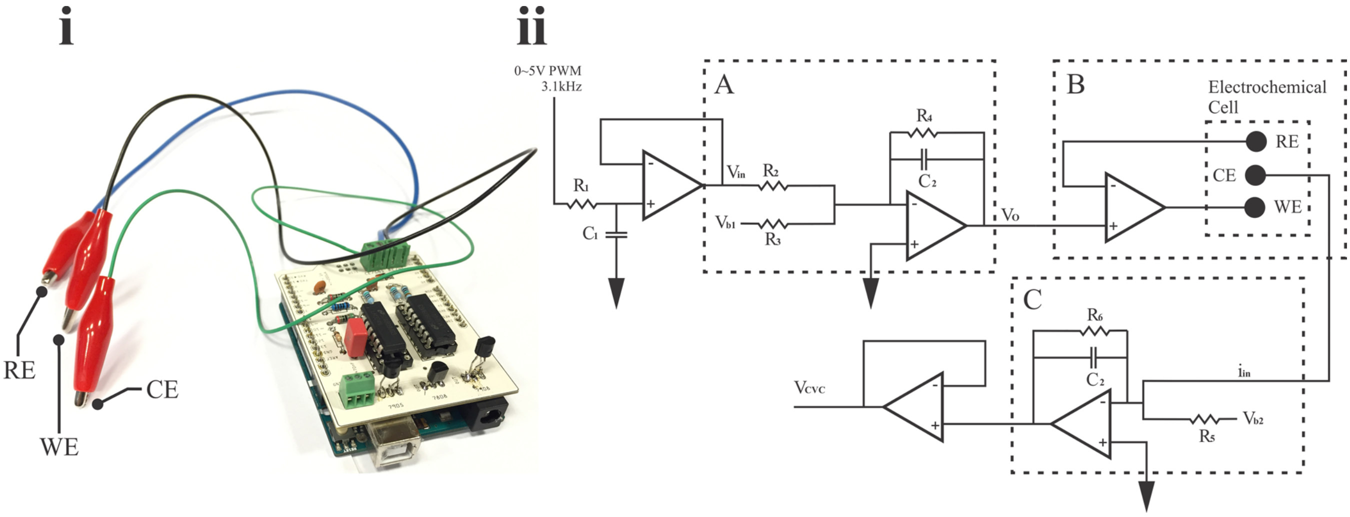

Figure 1. (i) Possible construction of the device using a custom etched printed circuit board. (ii) Analog circuit of the potentiostat with highlighted (A) summing amplifier; (B) electrochemical cell and electrochemical cell potential controlling amplifier; and (C) transimpedence amplifier (current to voltage converter). See Supporting Information for the values and the relationship between the components R_1 to R_6 and C_1 and C_2.

The proposed device uses an Arduino microcontroller board (Arduino Uno, Arduino) for parameter control and data acquisition. The Uno board runs a basic sketch (see Supporting Information) that enables the user to perform simple cyclic voltammetry (CV) experiments by choosing the start potential, vertex potential (inflection on the E vs t curve), and scan rate. The script can be easily understood and changed to perform different experiments (such as chronoamperometry), making the platform extremely versatile. The device potential window and current limits are defined by the summing amplifier (Figure 1ii(A)) and the transimpedence amplifier (Figure 1ii(C)), respectively. These are further explained in the Supporting Information.

Resistors Polarization Curve

To assess the capabilities of the fabricated equipment and to verify if it was working as expected, a 1 and 10 kΩ resistor were used. There resistors were connected between the working electrode and the counter and reference electrode (which were short-circuited together). The well-behaved and well-known response of these electronic compounds when submitted to a potential ramp make it easy to evaluate the performance of the equipment. When a potential is swapped between the leads of a resistor, the measured current is expected to show a linear response with a slope that corresponds to the inverse of the resistance value of the resistor ($1/R$). As seen in Figure 2, the expected behavior is observed and the resistances can be calculated as being 1044.9 ± 0.4, 10584.5 ± 19.0, and 5273.7 ± 4.5 Ω for the 1, 10, and 5 kΩ parallel resistor association, respectively. These values are in good agreement with the 10% tolerance resistor used.

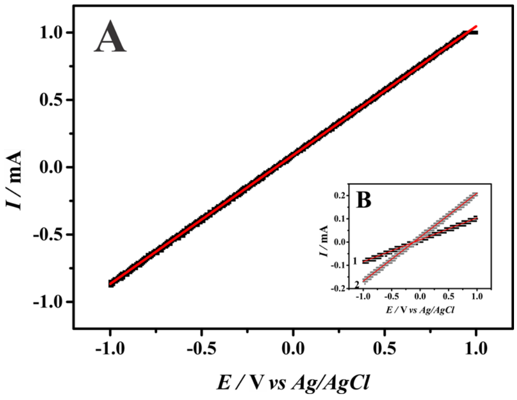

Figure 2. Dots: Polarization curves for (A) 1 kΩ resistor, (B1) 10 kΩ resistor, and (B2) 5 kΩ parallel resistor association. Line: Linear fit for the obtained curves with R^2 = 0.9996, 0.9984, and 0.9933 for 1, 5, and 10 kΩ, respectively.

Diffusion Coefficient Determination

To prove the capabilities of the potentiostat, a basic electrochemical experiment based on the redox reaction of potassium ferricyanide salt in water was performed. The diffusion coefficient of potassium ferricyanide was calculated by recording CV experiments in a solution containing a known concentration of the salt for different scan rates and using the Randles-Sevick equation (see Supporting Information). Although simple, this experiment is widely used as it illustrates the important electrochemical aspects and parameters[9] to students dealing with electrochemistry or CV for the first time. The values of all the components used for the setup during the electrochemical experiments are given in detail in Figure S2 and Table S1. A platinum working electrode with a diameter of 1.6 mm was used in a conventional three-electrode electrochemical cell. An Ag/AgCl wire was used as a quasi-reference electrode and a platinum wire as a counter electrode. The electrochemical cell was filled with 19.35 mM potassium ferricyanide in 0.1 M KCl solution. The potential was swapped between 1 and −1 V with scan rates of 0.01, 0.02, 0.05, 0.10, 0.20, 0.25, and 0.3 V s^{-1}. The recorded data can be seen in Figure 3.

As expected, Figure 3 shows an increase in the recorded electrochemical current with an increase in the scan rate. Figure 3B shows the plot of the anodic peak current vs the square root of the scan rate. As expected, the plot is a line and a linear fit with an R^2 of 0.9993 is obtained. From the slope of the linear fit and Randles-Sevick equation, the diffusion coefficient of potassium ferricyanide is calculated to be $(6.93 \pm 0.07) \times 10^{-6}$ cm^2 s^{-1}, which is in good agreement with the value in the literature.[10]

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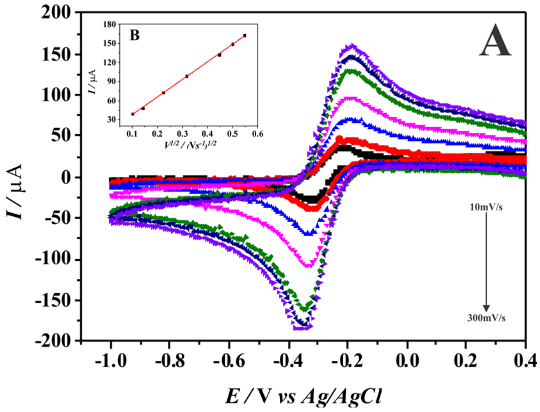

Figure 3. Cyclic voltammogram recorded on a platinum working electrode in 19.35 mM potassium ferricyanide in 0.1 M potassium chloride solution with scan rates of 0.01, 0.02, 0.05, 0.10, 0.20, 0.25, and 0.30 V s^{-1}. (Inset B) Linear fit of the anodic peak currents against the square root of the scan rate.

CONCLUSION

It is demonstrated that the fabrication and design of a simple potentiostat that is capable of resolving a current of the order of a few microamperes (μA) is possible using no more than a handful of simple and readily available electronic components. The simple design makes it possible for students with a basic electronics background to understand how a potentiostat operates and to recognize the equipment limitations and sources of error. The proposed device can be applied in joint teaching experiments encompassing electrochemistry, electronics, and programming. Although somewhat limited, especially when compared with commercial equipment, the fact that the proposed device costs less than $30.00 in materials (including the Arduino Uno board) makes it a competitive alternative for places where electrochemistry experiments take place on computer software or web based simulators.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.5b00961.

> Complete electronic diagram, DAC and ADC truth tables, and the source code for the potentiostat (PDF, DOCX)

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Notes

The authors declare no competing financial interest.

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