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A VERSATILE POTENTIOSTAT WITH OPTIONAL COMPUTER CONTROL

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ABSTRACT

A versatile potentiostat which can supply a maximum of 125 ma is described. The potentiostat uses readily available electronic components and an interface is detailed which allows the potentiostat optional computer control.

INTRODUCTION

Countless articles describing applications and circuit designs for three electrode potentiostats have appeared since introduction of this potential control technique in the late fifties.

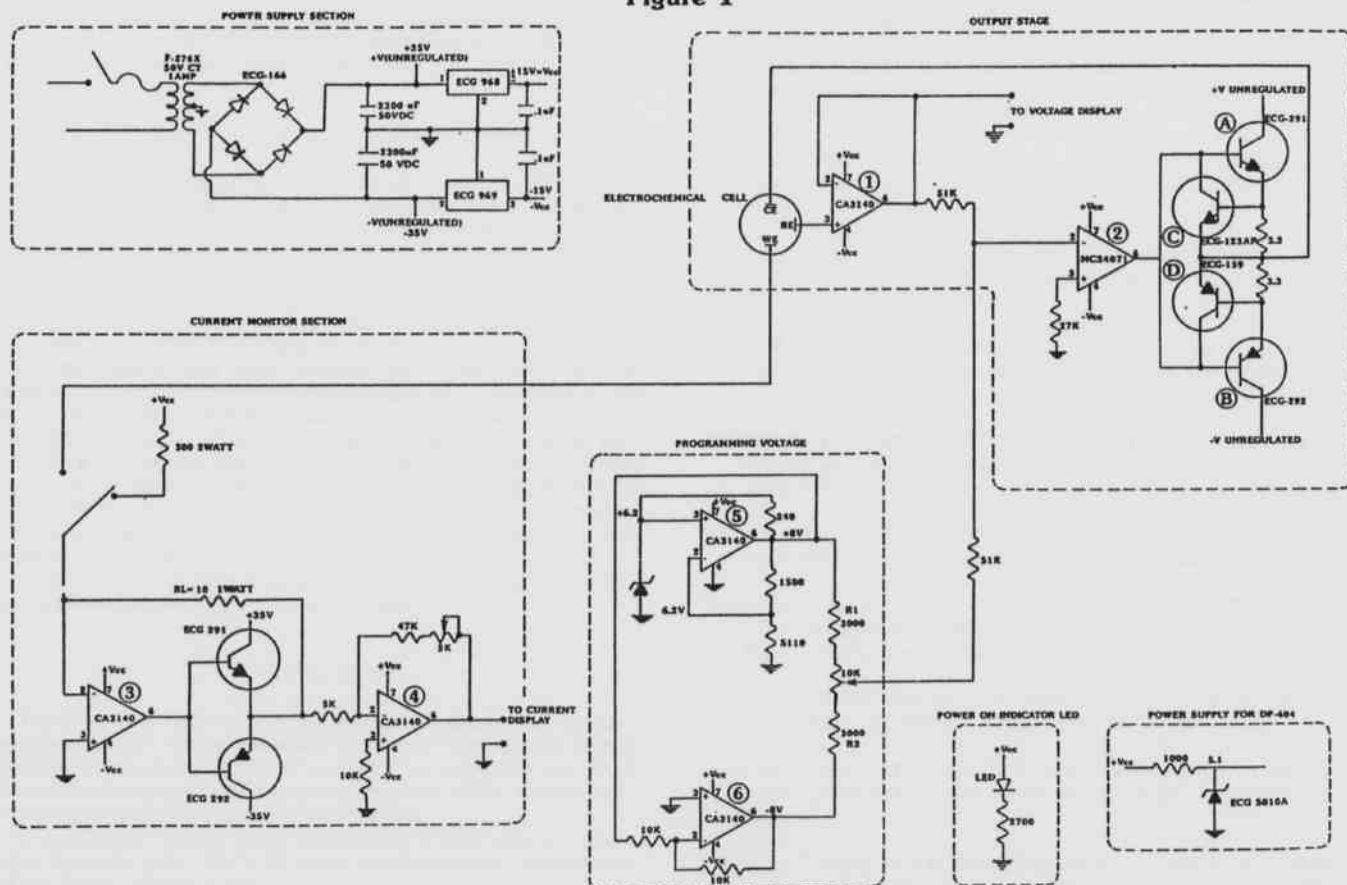
The basic function of a potentiostat is to maintain the potential of a working electrode (WE) at a desired fixed value, or to control that potential under a defined functional variation with respect to a reference electrode (RE). To perform a simple electrolysis under constant potential conditions, the working electrode voltage must be maintained at a fixed value. This constant potential should be maintained regardless of any fluctuations of electrolytes, current, solution resistance, temperature, or other factors. The potential of the WE is measured against a RE when the RE is placed very near to the WE via an apparatus such as a luggin capillary (Skoog, 1985). The desired potential is preset into the potential control circuit either manually or by computer control. The voltage difference, E (WE-RE), is sensed and

balanced against this preset potential. The difference or error signal is fed back into a control amplifier which varies the output of the control amplifier as necessary to maintain the preset potential. The apparatus thus will reduce or increase the cell current and also will follow possible changes of the cell resistance as the electroactive species are consumed. Essentially no current flows through the RE circuit, and its potential, therefore, remains stable because of the extremely high input impedance ($> 10^{11}$ ohms) of the electrometer operational amplifier used to sense the potential of the reference electrode.

CIRCUIT DESCRIPTION

The described potentiostat is a versatile high precision instrument capable of controlling a range of potentials (± 5 volts at the reference electrode) at a maximum current of 125 ma for application to a wide variety of electrochemical experiments.

Figure 1



The heart of the potentiostat is the two operational amplifiers and transistor network as shown in the output stage of Fig. 1. Amplifier 1 is a FET input amplifier which buffers the reference electrode voltage by minimizing any reference electrode current. Amplifier 2 is used to sum the constant voltage set by the programming voltage section and the voltage sensed by the reference electrode. As shown, the electrochemical cell completes the feedback path and any voltage changes at the reference electrode are coupled to the inverting input of amplifier 2 such that its output varies the electrochemical cell current via the two transistor (A and B) push-pull current booster. Thus, the controlled current output maintains a constant potential between the working electrode and the reference electrode. Transistors C and D were added to limit the cell current. If the user needs more current, a Darlington arrangement could be incorporated. A high voltage amplifier (amplifier 2) was used to increase the cell potential range.

In order to select a reference voltage, a stable source is required. Amplifiers 5 and 6 in the programming voltage section use a constant current configuration and a Zener diode's voltage stability to provide a stable input voltage for amplifier 5 and subsequently amplifier 2. A 10 turn potentiometer is used to vary the programming voltage. Resistors R1 and R2 can be adjusted to change the reference voltage range.

The second part of the circuit is the current monitor. Operational amplifier 3 and the two transistors attached to the output (pin 6), and RL (10 Ω, 1 watt) form a transresistance amplifier, better known as a current-to-voltage converter. The two transistors provide extra output current for the operational amplifier which is limited to a maximum of 12 ma. The input (pin 2) of operational amplifier 3 is forced to remain at ground potential by virtue of negative feedback. Any current flowing into the circuit is balanced by current leaving the input through RL; therefore, the voltage of the output of the transresistance amplifier is proportional to the input current which is the total current through the electrochemical cell. The resultant output voltage for amplifier 3 follows the relationship: $V_{out} = (I_{cell}) * RL$. The output of the transresistance amplifier is inverted and scaled by the inverting amplifier 4.

A double pole-double throw switch was added in the current monitor section. The double pole-double throw switch selects one of two circuit modes; current calibration or potentiostat operation. In the current calibration mode, a known current passes through the 300 ohm precision resistor to the current monitor circuit. A 5 kilo ohm 10 turn potentiometer is adjusted to give the correct voltage output to the current display.

Two digital panel meters are required to monitor the reference voltage and the electrochemical cell current. The reference voltage was monitored at the output of the buffer amplifier 1 and the total cell current was monitored at the output of amplifier 4.

COMPUTER CONTROL FOR THE POTENTIOSTAT

A computer controlled digital-to-analog converter was added to the potentiostat to provide greater flexibility. A 12-bit National Semiconductor binary multiplying digital to analog converter DAC1222 (DAC) was chosen to provide the conversions. The 12-bit DAC allowed a resolution of 0.00244 volts per step over the chosen +5 to -5 voltage range. An interface was constructed between the computer and the DAC, as shown in Fig. 2, which consisted of an address decode and various data latches to support the DAC.

The interface between the computer and the DAC was designed so that it could be used with other IBM compatible computers. An IBM XT-compatible was used in this laboratory. The DAC interface required ± 15 volts at approximately 20 ma and +5 volts at approximately 130 ma. The ± 15 volts were provided by the potentiostat power supply section and the +5 volts were provided by the computer.

Output voltage data were taken directly off the data bus and loaded into two octal data latches (74LS373), one at a time. After both latches are loaded, a second pair of latches are pulsed and the voltage data are transferred to the DAC for conversion. Care was taken to ensure that the latches were in close proximity to the data bus. Small distances (less than one foot) will ensure clean, valid output voltage

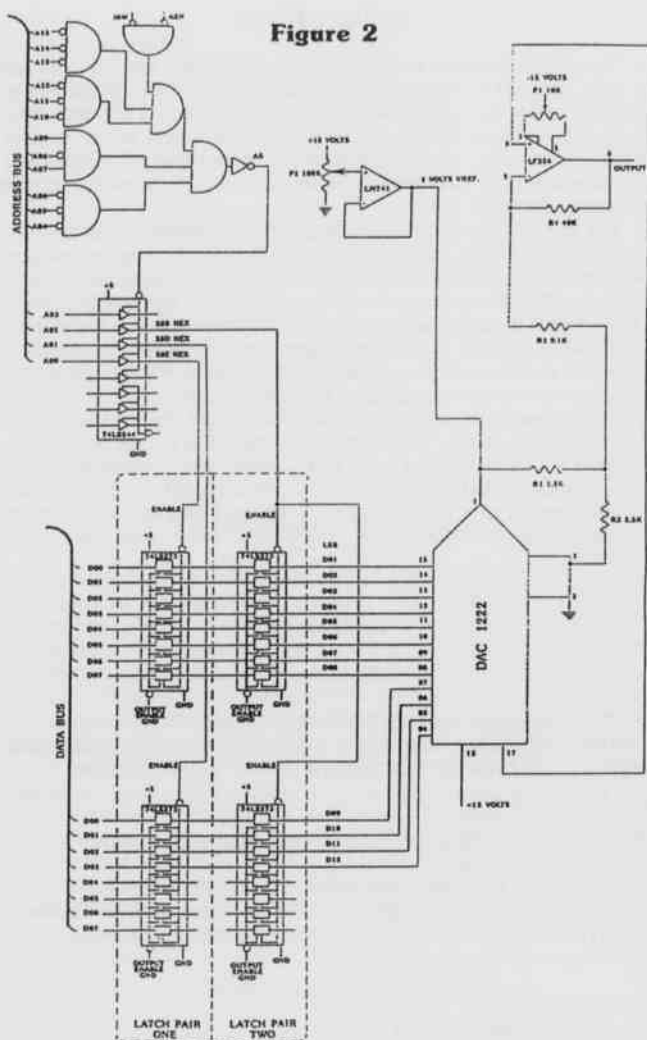


Figure 2

data to the latches. The first pair of latches was used to change the 8-bit data bus of the computer to 12-bits required by the DAC. A second set of latches was used in this design since the DAC did not have an enable line for conversion initiation. Thus, the voltage data (all 12-bits) could be transferred to the DAC simultaneously permitting the DAC to convert the correct voltage data instead of two pieces of data. Two allow the 12-bit DAC bipolar voltage swings, a LF356 JFET input OP-AMP was used (Fig. 2). Equations for the resistors are (National Semiconductor Corp., 1982):

$$AV = \text{Peak Voltage out} / \text{Voltage reference}$$

$$R4 = (2 AV - 1) R$$

$$R1 = R2 (AV - 1) / AV$$

$$R3 = R1 :: R2,$$

where R = 10,000 ohms, R2 = 2500 ohms, and the voltage reference = 2 volts.

Potentiometers were used for the resistance values to allow final adjustment of the DAC to eliminate any offsets caused by varying component tolerances. Resistor R2 was used to zero tolerance differences found in the linear op-amp while resistor R4 was used to adjust the ± peak voltages in the design. Resistor R3 was found to be the most critical in allowed tolerances.

A basic program to control the potentiostat is included in Appendix 1.

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APPLICATIONS

This design provides a versatile instrument which may be used for a wide range of electrochemical applications. It is the intent of the authors to use the potentiostat as a teaching tool in a graduate level course in electrochemistry. In this role the instrument will be used to plate copper from solution, demonstrate electrolysis of solvents and ion migration, and generally familiarize students with the various aspects of electrochemical systems.

The current application of concern to the authors is the anodic oxidation of PCB's. It has been shown that PCB's may be oxidized at a ruthenium dioxide anode (Laule *et al.*, 1986). This initial success with 4,4' PCB has prompted work to optimize the reaction conditions. The reaction is carried out on a 5 ppm solution of PCB in a 50/50 acetic acid/water solution. An oxidizing agent such as potassium persulfate is added and the solution subjected to a potential of 2.00 volts versus a silver/silver chloride reference electrode for 48 hours.

The present work is an attempt to optimize the yield of the reaction at room temperature. The variables of concern are: time of reaction, applied potential, oxidant used, and exposure to UV radiation. Initial results indicate that the exposure of the reaction cell to UV light greatly enhances the yield of the reaction. Not enough data have been received to make any predictions as to the effect of other conditions on the reaction.

APPENDIX 1

```

'
' Computer Control for the Potentiostat 10-29-88
'THIS PROGRAM WRITTEN FOR THE QUICKBASIC COM-
'PILER. IT CAN BE MODIFIED TO FIT YOUR NEEDS.

```

- ```

' 1) Voltage entered (+5 to -5 volts) and converted to positive
' voltages only.
' 2) Voltage is converted to its decimal equivalent voltage 0.0024414
' volts per step
' 3) Voltage is converted to binary form " 1111111111 "
' 4) The high four and low eight are converted back to decimal
' 5) Voltage data loaded into the latches and transferred to DAC

```

```
start:
```

```

input " enter voltage ",volt
volt = volt + 5.00 :changes the voltage to positive only
volt = cint (volt/.0024414)
print "decimal convert volt ",volt

```

```

'
' converts voltage to binary
'

```

```

x = 2048:bin$ = ""
convert:
volt1 = (volt - x)
if volt1 >= 0 then bin$ = bin$ + "1"
if volt1 < 0 then bin$ = bin$ + "0"
if volt1 >= 0 then volt = volt1
x = x / 2
if x < 1 goto continue
goto convert

```

```

'-----
continue:

```

```

'-----
'
' after conversion voltage is in form " 1111111111 "
' low$ will take the right 8 bits for loading into latches
' high$ will take the left four for loading into latches
'-----

```

```

low$ = right$(bin$,8)
high$ = left$(bin$,4)

```

```

'-----
' convert low binary to decimal.
' conversion fo the lower 8 bits back to decimal for output to
' the latches
'-----

```

```

i1 = 0 : bits = 1
for x = 1 to 8
shift$ = right$(low$,x): shift$ = left$(shift$,1)
if shift$ = "1" then i1 = i1 + bits
bits = bits * 2
next x
print "lowload ";i1;

```

```

'-----
' convert high binary to decimal
' conversion of the high 4 bits into decimal for loading into the
' latches
'-----

```

```

i2 = 0 : bits = 1
for x = 1 to 4
shift$ = right$(high$,x)
shift$ = left$(shift$,1)
if shift$ = "1" then i2 = i2 + bits
bits = bits * 2
next x
print "high load ";i2;

```

```

'-----
' the loading of the latches
' 654 dec = 28E hex load latch one
' 653 dec = 28D hex load latch two
' 651 dec = 28B hex strobe data to dat
'-----

```

```

print "loading the d / a ", q
out 654,11 ;'load one (1)
out 653,12 ;'load two (2)
print "output enabled " ,q
out 651,0 ;'output enable
goto start
end

```

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