Operating Instructions
for the
AFCBP1 Bipotentiostat

from
Pine Instrument Company
Grove City, Pennsylvania

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Introduction

The AFCBP1 bipotentiostat is a general purpose electrochemical instrument with a built-in analog voltage Sweep Generator. It can function as a traditional potentiostat or galvanostat using three electrodes (working, reference, and counter electrodes), and it may also independently control an additional (second) working electrode. While this "bi"-potentiostat feature was originally designed for use in classic ring-disk electrode voltammetry, the instrument has also found use in many other dual working electrode techniques.

Features

Multi-Purpose Potentiostat and Galvanostat

The AFCBP1 can be operated as either a three or four electrode potentiostat for use in many kinds of electrochemical experiments, including cyclic voltammetry, anodic stripping voltammetry, rotated disk voltammetry, rotated ring-disk voltammetry, and interdigitized array experiments. It can also be operated as a two-electrode device for tracing the current-voltage characteristics of electrochemical systems. One of the working electrodes can be operated in a galvanostatic manner where the current is controlled.

Analog Sweep Generator

The dual range Sweep Generator produces a true analog voltage sweep. The user controls the sweep rate and limits rather than the period or frequency of the sweep. Particular attention has been applied to the circuit design to provide flexibility in adjusting, sweeping, or stopping the sweep at any point in a particular cycle. Its output may also be used independently for non-electrochemical purposes. Sweep rate and limits are set using push button potentiometers.

Flexible Voltage Control

The AFCBP1 offers independent control of the potential at each working electrode. The applied potential may reflect any combination of the Sweep Generator output, an offset voltage adjusted on the front panel, or an external voltage source. Summing circuits add these three inputs and apply the sum to the electrode. The electrode voltage is presented on the front panel. Offset voltages are set using push button potentiometers.

Current Conversion

The current through each working electrode is presented as a proportional voltage on a front panel output. This voltage signal is suitable for driving an XY Recorder or sampling by a digital oscilloscope or computer controlled A-to-D board. Each working electrode has seven decades current-to-voltage conversions, easily selected using push buttons on the front panel.

Front Panel Voltmeter
A 3-1/2 digit voltmeter displays voltages for all principle front panel outputs.

Overload Indicator
Saturation of any internal amplifiers is signaled by a front panel LED.

Front Panel Connections
All connections are made via banana jacks except the reference electrode input, which utilizes a BNC type shielded connector. Signal outputs are protected against short circuits to circuit common.

Dummy Cell
A set of internal resistors can be switched in place of the external electrochemical cell, providing a way to idle the instrument between experiments.

Warranty

The AFCBP1 manufactured by Pine Instrument Company is warranted to be free from defects in material and workmanship for a 6 month period from date of shipment to original purchaser and used under normal conditions. The obligation under this warranty being limited to replacing or repairing any part or parts which shall upon examination disclose to Pine Instrument's satisfaction to have been defective and shall have been returned freight pre-paid and clear of encumbrances to Pine Instrument Company in Grove City, PA within the warranty period. This warranty being expressly in lieu of all other warranties expressed or implied and all other obligations or liabilities. All specifications are subject to change without notice.

Scope of this Manual

This manual focuses on the use of the AFCBP1 as a "stand-alone" instrument. The user is assumed to have some type of recording device, such as an XY plotter or digital oscilloscope, connected to the instrument. Further, it is assumed that the user is familiar with the subject of electrochemistry, especially cyclic voltammetry.

Users who primarily control the AFCBP1 via the AKCBP1 software package may still find this manual useful as it describes the capabilities of the instrument in detail. Those who are interested in upgrading their system to a computer controlled instrument should contact Pine Instrument Company for more information.

Setting Up the System

Inspection
The AFCBP1 Potentiostat should be inspected for shipping damage immediately after unpacking. If any damage is found, please notify the carrier and Pine Instrument Company as soon as possible.

Installation

The AFCBP1 has a rugged design and is intended for laboratory use. Exposure to corrosive chemicals, however, may damage the exterior finish on the chassis. Special care should also be given to the markings on the non-metal surface of the front panel.

The instrument may be placed on a suitable desk, lab bench, or equipment rack. Access to the rear panel is rarely required in normal use, but the unit should be positioned so that all front panel controls and connections are readily accessible. Connections between the AFCBP1 and external recording devices should be made using shielded coaxial cables whenever possible. If the AFCBP1 is to be connected to a computer, careful thought should be given to the placement of the computer and the cable as well.

Setting the Proper Line Input Voltage

The power entry module located on the rear panel is where AC voltage enters the AFCBP1 bipotentiostat. The module features a power cord receptacle that allows for easy removal or changing of the power cord. There is a line fuse to help protect the bipotentiostat's internal circuitry, and there is also an input voltage selection system that allows selection of either 120 or 240 VAC as the input voltage.

The correct operating voltage is set with the voltage selector card inside the module. The "120" setting should be used for line voltages between 100 and 125 VAC. The "240" setting should be used for line voltages between 200 and 250 VAC. To change the factory setting, perform the following operations:

1)   Remove the power cord from the module.
2)   Slide the clear door to the left.
3)   Use a pair of pliers to remove the voltage selector card.
4)   Re-insert the card with the desired voltage visible (120 or 240 VAC).
5)   Plug the power cord back into the module.

Testing

If the AFCBP1 is not operating properly, always check the fuse first. The fuse is located in the power entry module on the back panel. Remove the power cord, slide the clear door to the left, and use the "PULL FUSE" lever to remove the fuse. If required, replace fuse with a 3 Amp Slow Blow type fuse after checking for the cause of the blown fuse. If the fuse is in good condition, the operation of the unit can be tested as outlined in the "Troubleshooting" section.

The Front Panel

Located within the AFCBP1 are a fully functional bipotentiostat and an independent analog sweep generator. Both of these electronic circuits are controlled by an internal microprocessor which, in turn, is controlled by your choice of settings on the front panel. Adjusting these settings is a simple matter, as all front panel controls (except one) are of the push button type, and forty-one small red indicator lights instantly reflect any adjustments that you make to these settings. In addition, the internal microprocessor is equipped with non-volatile memory so that the AFCBP1 "remembers" all of your front panel settings even when the power is off.

Controls and jacks are grouped according to their function into seven main sections on the front panel (see Figure 1). The three largest sections are labelled Sweep Generator, K1 Electrode, and K2 Electrode. The Voltmeter and other general controls are located on the left side of the panel. Cell connections are made using the four jacks at the right side of the panel. The rear panel has six jacks which allow connection of external frequency compensation circuitry, and there is also a fifty pin connector used to attach the AFCBP1 to a computer.

General Controls

Located in the lower left hand corner of the front panel are a group of controls that affect the general operation of the AFCBP1 (see Figure 2). When the power switch is switched on, the internal power supply supplies power to all internal circuits. When the power switch is switched off, an internal battery (with a 10-year lifetime) supplies power to the non-volatile memory used to store the front panel settings.

The CONTROL SOURCE push button setting determines whether the bipotentiostat is under the control of the front panel or an external computer. This control must be switched to the "external" setting when using the AFCBP1 with an external computer. For stand-alone use, the control is switched to the "panel" setting. When in the "external" mode, all front panel settings are inactive and/or ignored except for the voltmeter control and the control source button itself. This allows you to switch between modes at any time. Note that this button has a one second delay in order to prevent accidental mode changes.

The two ELECTRODES controls determine how and if the bipotentiostat is connected to the external electrochemical cell. The OPEN control is normally deactivated, but can be activated if you wish to make "open loop potential" measurements using a pH or ion selective electrode (discussed later).
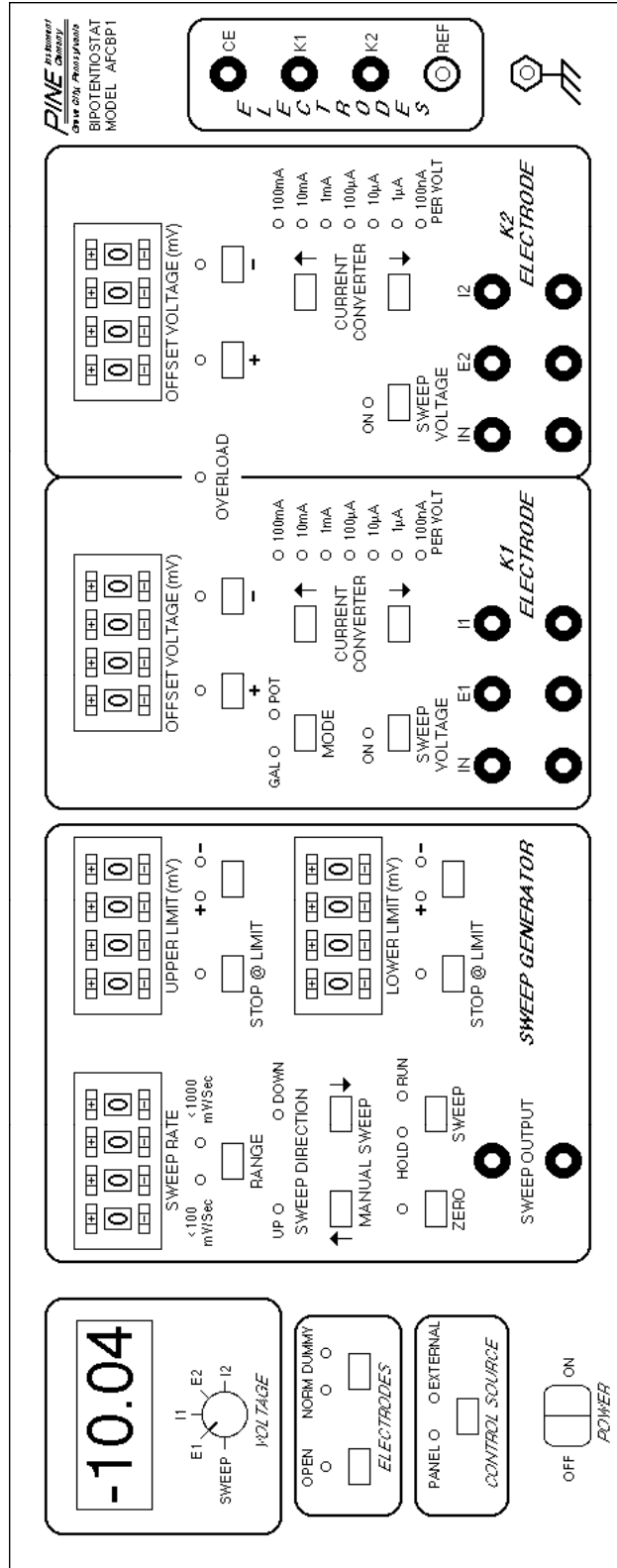
FIGURE 1: AFCBP1 FRONT PANEL ILLUSTRATION
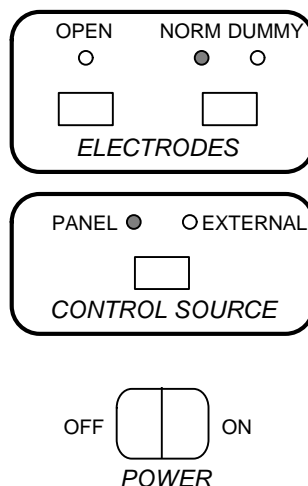
FIGURE 2: GENERAL CONTROLS

The NORM vs DUMMY control is used to switch the electrode connections between an internal resistor network (the dummy cell) and the (normal) external electrochemical cell. Usually, you switch to "dummy" mode while setting up an experiment and switch back to "normal" mode just before starting an experiment.

Electrode Connections

There are four jacks located on the far right side of the front panel that are used to make connections to the electrochemical cell (see Figure 3). The upper banana jack is a low impedance output to the auxiliary or counter electrode (CE).
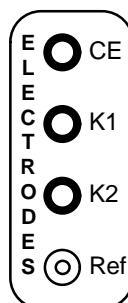
FIGURE 3: ELECTRODE CONNECTIONS

Below the CE jack are two jacks labelled K1 and K2 which are the connections to the working electrodes. If you are using a conventional three electrode system, the working electrode should be connected to the K1 jack (which is held at virtual DC common by feedback circuitry). If you are using a four electrode system, then connect the working electrode that is physically nearer to the reference electrode to the K1 circuit. For rotated ring-disk electrodes, the disk should be connected to K1 and the ring to K2.

The reference electrode connector (REF) is of the shielded BNC type. The reference electrode should be connected to the central (signal) connection on this jack. This is a high impedance input which measures the reference

electrode voltage without loading the system. The cable which connects the AFCBP1 to the reference electrode is susceptible to picking up stray electrical noise.

To help prevent noise problems, a coaxial cable should be used to connect the reference electrode. The outside (shielding) connection on the BNC jack provides a driven shield that should be connected to the sheath of the coaxial cable. No connection should be made to this sheath at the electrochemical cell, and by no means should it be grounded.

Sweep Generator

The AFCBP1 has a built-in, true analog voltage sweep generator capable of providing the potential sweep signal required for most cyclic voltammetry experiments. The output of this dual range Sweep Generator may be used to supply a sweep signal to either or both working electrodes and is also available at the front panel for other uses. Particular attention has been given to the design of the Sweep Generator circuit so that there is complete flexibility in adjusting or stopping the sweep at any point in the sweep cycle. All Sweep Generator settings are indicated by over a dozen front panel lights, and easy-to-use push button controls allow these settings to be adjusted at any time.

The Sweep Generator does not operate by controlling the period or frequency of a "triangle" wave, and neither does it generate a digital "stairstep" like other popular sweep generators. The three controlled parameters for this analog sweep are its rate, the upper sweep limit, and the lower sweep limit. All three of these parameters are adjusted using four-digit push button potentiometers. Other push button controls allow the sweep direction to be adjusted "on-the-fly" and control how and at what potential the sweep is stopped.

In a repetitive sweep, the upper and lower limits of sweep are independently adjustable between +9.999 volts and -9.999 volts. The output signal sweeps between the high and low limits at the set sweep rate. The sweep can be stopped and held at any point, then started in either direction from the stop point at the discretion of the operator. In a normal operation, the Sweep Generator is free running. If desired, the Sweep Generator can be set to automatically stop at either end of the sweep. The voltage will hold at that point until the sweep circuit is started again.

Each of the Sweep Generator functions is discussed in more detail below. The user is encouraged to spend time experimenting with these controls. The built-in Voltmeter can be used to monitor the sweep output during such experimentation. With a little practice, one can easily become adept at producing just about any desired sweep signal for cyclic voltammetry.

Using the Sweep Output

The Sweep Generator is a self-contained, independent circuit within the AFCBP1, and the sweep output may be used as the signal for a cyclic voltammetry experiment or for some other non-electrochemical application. In the latter case, an external connection can be made to two output jacks on the front panel labelled SWEEP OUTPUT. This output may also find use as a trigger in some applications.

Usually, however, you will want the output of the sweep generator to be applied to the K1 and/or K2 working electrode circuit. This connection is made internally by activating the SWEEP VOLTAGE control in the appropriate electrode control section on the front panel.

Adjusting the Sweep Limits

In most voltammetry experiments, it is desirable to sweep the working electrode potential one or more times between two "limit" potentials. One of these, the upper limit must always be set at a more positive potential than the lower limit. The Sweep Generator has two identical sets of controls for adjusting the upper limit and lower limit. These are shown side-by-side in Figure 4, but on the front panel they are on top of each other.
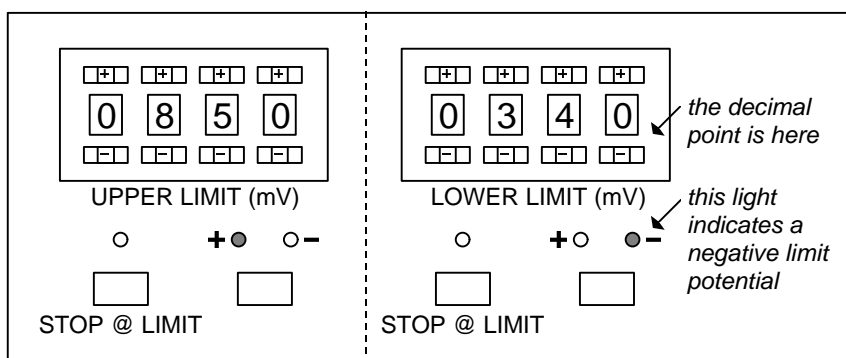
FIGURE 4: THE SWEEP LIMIT CONTROLS

The four-digit push button potentiometers permit individual setting of each numeric digit in the limit potential using the small buttons above and below each digit. You can set any number of millivolts between "0000" and "9999". Note that the decimal point is located immediately to the right of the fourth digit.

Just below each push button potentiometer are two push buttons. The one labelled STOP @ LIMIT is discussed later, while the other (unlabeled) push button is used to change the mathematical sign on the limit potential. Two small lights above this push button indicate whether the sign is positive (+) or negative (−). In Figure 4, the upper limit is set at +850 millivolts, and the lower limit is set at −340 millivolts. The limit potentials can be set both positive, both negative, or one positive and one negative; however, the upper limit must always be more positive than the lower limit. The absolute difference between the two limits should exceed ten millivolts.

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Adjusting the Sweep Rate

There are two sweep rate ranges available using the front panel controls. The slower range operates between 0.00 and 99.99 mV/sec while the faster range operates between 000.0 and 999.9 mV/sec. (Under external computer control, sweep rates up to 10,000 mV/sec are available.) The desired range and sweep rate are adjusted using the controls shown in Figure 5.

The four-digit push button potentiometers permit individual setting of each numeric digit in the limit potential using the small buttons above and below each digit. You can set any number between "0000" and "9999", and the sweep rate is read from the panel in millivolts per second. Note that the position of the decimal point depends on the range setting, as illustrated in Figure 5. Two small indicator lights show which range setting is presently chosen.
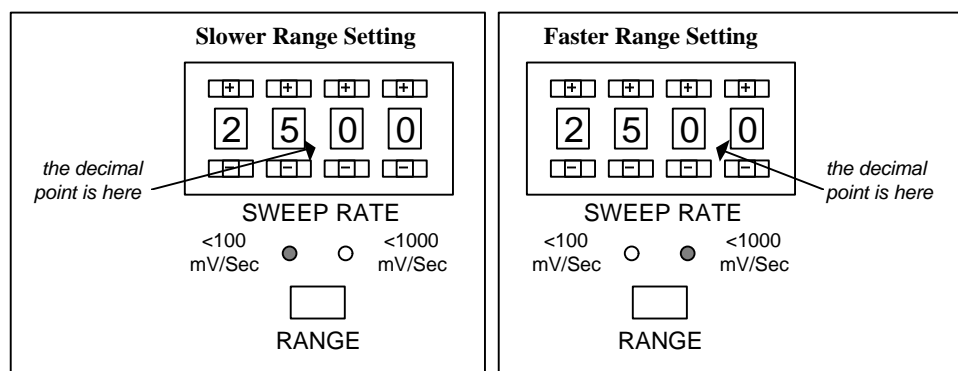
FIGURE 5: THE DUAL RANGE SWEEP RATE CONTROLS

Adjusting the Initial Potential

The next three sections describe how to adjust the sweep output to just about any initial potential. While you are adjusting the initial potential, however, it is possible that the potential may make an excursion through a range where significant electrochemical activity occurs. To prevent this, it is a very good idea to put the bipotentiostat in "dummy" mode while adjusting the potential, returning it to normal mode only after the desired initial potential has been set up.
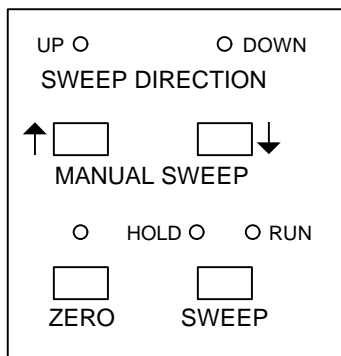
FIGURE 6: THE SWEEP CONTROLS

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Zeroing the Sweep Output

A section of the Sweep Generator control panel containing four push buttons is shown in Figure 6. These controls are used together to adjust the sweep output to any desired potential. To zero the sweep output, simply activate the ZERO control, which overrides all other settings and sends the sweep output to zero volts and holds it there until deactivated.

At this point, you can switch the Sweep Generator to "hold" mode using the SWEEP control. This allows you to deactivate the zero control while maintaining the sweep output at zero volts. This is the normal starting point for many of the adjustments described below.

Manual Adjustment of the Sweep Output

Once you have set up the sweep limits and the sweep rate, you may wish to adjust the sweep output to some arbitrary potential. You should begin by setting the sweep output to zero volts as described above. Then, you can manually move the sweep output up or down using the MANUAL SWEEP buttons. The SWEEP control should be in "hold" mode while making this kind of adjustment.

When one of the MANUAL SWEEP buttons is pressed, the sweep output moves in the appropriate direction at the present sweep rate. By watching the front panel voltmeter carefully, the sweep output may be adjusted to any desired value. If the value changes too quickly for you to make a precise adjustment, you should temporarily set the sweep rate to a lower value.

Note that pressing either MANUAL SWEEP button usually changes the sweep direction, as indicated by the two small indicator lights located just above the buttons. After making a manual adjustment and before starting the sweep, make certain that the sweep direction is set to the direction you want it to go. Also note that if you adjust the potential to some value outside the range bracketed by the upper and lower limit potentials, the sweep direction is automatically set to head back towards the bracketed range.

Moving the Sweep Output to a Limit Potential

Once you have set up the sweep limits and the sweep rate, you may desire to set the sweep output potential equal to one of the two limit potentials. While this can be done using the manual method described above, the method below is somewhat more convenient.

Begin by setting the sweep output to zero volts as described above, making certain that the SWEEP control is left in "hold" mode after zeroing the potential. Next, you should manually move the sweep output to some value that is in between the upper and lower limit potentials. (In most cases, this step is not actually required because zero will fall between the two limit potentials.)

Once in between the limit potentials, use a MANUAL SWEEP button to set the sweep direction towards the appropriate limit potential, and activate the appropriate STOP @ LIMIT button. If you wish to set the initial potential equal to

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the lower limit, then set the sweep direction to negative (down) and activate the lower STOP @ LIMIT button. If you wish to set the initial potential equal to the upper limit, then set the sweep direction to positive (up) and activate the upper STOP @ LIMIT button.

Next, turn on the sweep by switching the SWEEP control to "run" mode. The sweep output will move (at the present sweep rate) towards the desired limit potential, and then it will stop at that limit. At this point, switch the SWEEP control to "hold" mode and deactivate the STOP @ LIMIT control. The sweep output will hold at the limit potential until you decide to start the sweep.

Starting and Stopping the Sweep

The SWEEP control allows you to start and stop the sweep "on-the-fly" at any point during the sweep program. This push button switch alternately halts and then restarts the sweep. Two indicator lights associated with this switch indicate whether the sweep is in "hold" or "run" mode. Note that the ZERO control, described above, overrides the setting of the SWEEP control. That is, when the ZERO control is activated, the sweep output is sent to zero regardless of whether the Sweep Generator is in "hold" or "run" mode.

Setting the Sweep Direction

The two MANUAL SWEEP momentary push button controls are usually used to change the direction of a sweep in progress. When the left-hand "up" button is pressed, the voltage sweep proceeds in a positive direction. When the right-hand "down" button is pressed, the sweep proceeds in a negative direction. Sweep direction can be adjusted before or during a sweep, and two indicator lights above these push buttons indicate the present direction of the sweep. These two push buttons can also be used to adjust the Sweep Generator output when it is in "hold" mode.

Stopping at a Limit Potential

If the STOP @ LIMIT control associated with one of the limit potentials is activated, a potential sweep in progress is halted just as soon as it reaches that limit potential. Generally, you will use this control "on-the-fly" to stop the sweep at the end of an experiment. The precise behavior of the STOP @ LIMIT controls can be complex under unusual circumstances. There are three distinct conditions that influence how the upper STOP @ LIMIT control behaves:

- If the sweep output is less than the upper limit **and** the sweep direction is positive, then the sweep stops as soon as it hits the limit potential. (normal case)
- If the sweep output is less than the upper limit **and** the sweep direction is negative, then the sweep is halted immediately.
- If the sweep output is already greater than the upper limit potential (an unusual event), then the sweep is halted immediately.

Similar behavior can be expected from the **lower** stop @ limit switch. Note that if both STOP @ LIMIT controls are activated simultaneously, the sweep halts until one of the two controls is deactivated.

Electrode Controls

Both working electrodes (K1 and K2) operate independently of each other, and both have independent controls on the front panel. These controls allow you to specify the potential that is applied to a working electrode and its current sensitivity. Because both working electrodes have virtually identical controls, the descriptions given in this section of the manual apply equally to either the K1 or the K2 electrode controls. Note, however, that the K1 electrode has an additional control that permits it to be operated in a galvanostatic rather than a potentiostatic manner.

Applying an Offset Potential

An offset (or baseline) potential can be applied to a working electrode using a four-digit push button potentiometer to set any number of millivolts between "0000" and "9999". Two other controls (located just below the potentiometer) are used to set the mathematical sign, "+" or "−", for the offset potential. If both of these controls are deactivated, then no offset potential is applied to the electrode. Figure 7 illustrates three possible settings for the offset potential.
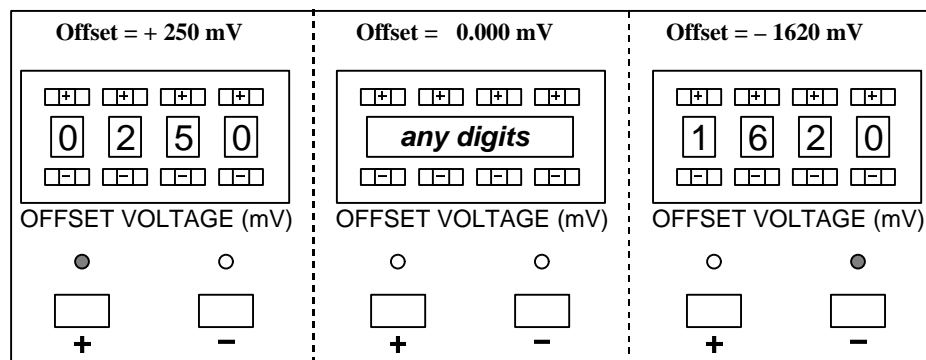
FIGURE 7: THE OFFSET VOLTAGE CONTROL

Use of the OFFSET VOLTAGE control is a convenient way to hold the potential of a working electrode at some non-zero value. For example, in rotated ring-disk voltammetry, one might sweep the potential of the disk electrode while holding the ring electrode at a some fixed potential set using the OFFSET VOLTAGE.

Another possible use for the OFFSET VOLTAGE control is to apply a sudden potential step to a working electrode, such as that required during a chronoamperometry experiment. A step from zero volts to some non-zero potential can be accomplished by starting with the OFFSET VOLTAGE deactivated and then manually depressing either the "+" or "−" push button to initiate the potential step to a non-zero potential.

Applying a Potential Sweep

If you wish to sweep the working electrode potential, you should activate the SWEEP VOLTAGE control. This applies the output of the Sweep Generator to the internal summing circuit for the working electrode. The indicator just above the SWEEP VOLTAGE control turns "on" whenever this control is active (see Figure 8).

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ON ⦿

☐
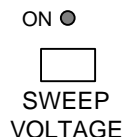
SWEEP
VOLTAGE

FIGURE 8: THE SWEEP VOLTAGE CONTROL

The Current Converter

The potentiostat controls the potential of a working electrode and measures the current passing through that electrode. The AFCBP1 features a current-to-voltage converting system that presents the working electrode current as a proportional voltage on a front panel output jack. This allows for easy connection to external recording devices, such as X-Y plotters or digital oscilloscopes.

The sensitivity of working electrode current measurements is set using the CURRENT CONVERTER control depicted in Figure 9. Seven decades of scaling factors, from 100 mA/V down to 100 nA/V, are available. Currents as low as 1 nA can be measured with the 100 nA/V setting and proper electrical shielding.

○ 100mA

☐ ↑   ○ 10mA

● 1mA

CURRENT   ○ 100µA
CONVERTER
○ 10µA

☐ ↓   ○ 1µA

○ 100nA
PER VOLT
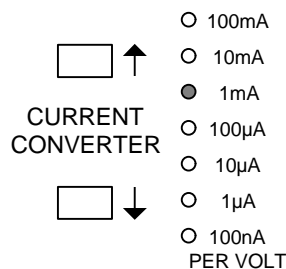
FIGURE 9: THE CURRENT CONVERTER

As an example, assume that the working electrode current is 5.72 mA. With the CURRENT CONVERTER set to 1 mA/V as shown in Figure 9, the signal presented at the front panel jack would be 5.72 volts. Obviously, it is a good idea to set the CURRENT CONVERTER to a range that closely matches the currents you expect to observe in a particular experiment. In general, the largest current that can be measured at any particular setting is about ten times the value listed next to the indicator light for that setting.

Signal Connections

Each working electrode section on the front panel has three pairs of banana jacks (see Figure 10). The E1 and I1 jacks are output signals suitable for driving XY plotters and digital oscilloscopes. The E1 jack presents the working electrode potential, and the I1 jack presents a voltage signal proportional to the working electrode current (see the discussion of the CURRENT CONVERTER control, above.)

IN   E1   I1
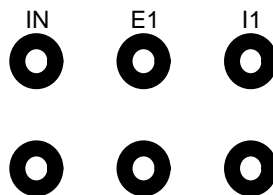
FIGURE 10:   K1 ELECTRODE SIGNAL JACKS

The IN jack is an input that is connected directly to the internal summing circuit for the working electrode. This jack is generally used to apply the output of some external function generator to the working electrode. For example, the AFCBP1 has no built-in provision for providing a pulse sequence for pulse polarography, but a third-party pulse programmer might provide this capability.

Voltmeter

A voltmeter is provided to help you measure various front panel signal voltages conveniently.   The actual reading displayed depends on the setting of the VOLTAGE knob control located under the voltmeter display.

To display the output of the Sweep Generator, turn the knob to the "sweep" position. The voltage presented across the SWEEP OUTPUT jacks is displayed on the voltmeter. Because the voltmeter display is only updated periodically, the display will tend to skip from one reading to the next in a discontinuous fashion during a potential sweep. This effect is most pronounced when using fast sweep rates.

To display the potential of a working electrode, turn the knob to either the "E1" or "E2" position. The voltmeter will display the appropriate potential corresponding to the potential being presented at the "E1" or "E2" output jacks on the front panel.

To monitor the current at a working electrode, put the knob in either the "I1" or "I2" position. The voltmeter will display a voltage which is proportional to the current at the electrode. The voltage is the same voltage which is presented across the front panel output jacks for "I1" or "I2". To convert this voltage to a current, multiply it by the setting of the appropriate CURRENT CONVERTER control.

Signal Connections

Most connections to the AFCBP1 are made via standard banana jacks located on the front panel. The reference electrode connection on the front panel, however, is of the BNC type to reduce noise. The only connections on the rear panel are six jacks for frequency compensation and the ribbon cable connector used for external computer control.

The SWEEP GENERATOR Section

SWEEP OUTPUT The Sweep Generator output is presented across this pair of jacks. It may be used independently of any other function of the AFCBP1.

The ELECTRODES Section

CE This red banana jack provides a low impedance output to the counter or auxiliary electrode.

K1 This yellow banana jack provides a low impedance connection to the disk or working electrode. Internal feedback circuitry holds this connection at virtual DC common.

K2 This blue banana jack provides a low impedance connection to the ring or second working electrode.

REF The reference electrode should be connected to the central (signal) connection on this BNC jack. This is a high impedance input which measures the reference electrode voltage without loading the system. The outside (shielding) connection connects to the sheath of a coaxial cable running to the reference electrode in the cell. No connection should be made to this sheath at the cell, and it should never be grounded.

Grounding Connections

DC COMMON All black jacks along the bottom of the front panel present the DC Common for the AFCBP1 circuit. They are not connected to either the chassis frame or the earth ground.

EARTH GROUND The Binding Post located near the lower right corner of the front panel is the chassis or earth ground. This is a convenient point for connecting the DC Common to the earth ground, if desired. This point represents earth ground only if the 3-prong power cord is connected to a properly grounded outlet.

The K1 ELECTRODE Section

IN This voltage input jack is connected through an internal 10K ohm resistor to the summing point for the K1 electrode circuitry. Usually an external function generator will be connected to this input. When the K1 electrode is operating in "Potentiostat" mode, this external voltage is summed with the OFFSET VOLTAGE and the SWEEP VOLTAGE (if activated) and then applied to the K1 electrode. When the K1 electrode is operated in "Galvanostat" mode, this voltage sum is converted to a current according to the CURRENT CONVERTER setting, and the resulting current is sent through the K1 working electrode.

E1 This voltage output jack provides a voltage indication of the actual K1 electrode voltage with respect to the REF electrode. This is a buffered output suitable for driving the input of an X-Y Recorder or for sampling by a digital oscilloscope or a computer controlled A-to-D board.

I1 This voltage output jack provides a voltage proportional to the actual K1 working electrode current. The CURRENT CONVERTER setting determines the factor used to convert current to voltage. For example, if 2.0 volts is presented at this jack, and the CURRENT CONVERTER is set at 10 mA per volt, then the actual K1 current is 20 mA. (i.e., 2.0 volts times 10 mA/V equals 20 mA.)

The K2 ELECTRODE Section

IN This voltage input jack is connected through an internal 10K ohm resistor to the summing point for the K2 electrode circuitry. Usually an external function generator will be connected to this input. The K2 electrode always operates in "Potentiostat" mode, and this external voltage is summed with the OFFSET VOLTAGE and the SWEEP VOLTAGE (if activated) and then applied to the K2 electrode.

E2 This voltage output jack provides a voltage indication of the actual K2 electrode voltage with respect to the REF electrode. This is a buffered output suitable for driving the input of an XY Recorder or for sampling by a digital oscilloscope or a computer controlled A-to-D board.

I2 This voltage output jack provides a voltage proportional to the actual K2 working electrode current. The CURRENT CONVERTER setting determines the factor used to convert current to voltage. See the description of I1, above.

REAR PANEL Connections

K1   These jacks, which operate in pairs, allow capacitors to be inserted into the
K2   AFCBP1 circuit to slow its response time. Oscillations which may occur
GAL under certain conditions can be eliminated in this way. Refer to the
         section on noise and oscillations for details.

EXTERN A fifty pin ribbon cable connector that allows external control of the
            AFCBP1 by an external computer equipped with an appropriate analog-to-
            digital interface board. See cable pinout at end of manual for futher
            details.

Instrument Controls

There are forty-one (41) small red light emitting diodes (LEDs) on the front panel that indicate the present state of the bipotentiostat. Most of these LEDs are associated with nearby switches and indicate the present setting of a particular switch. There is one LED, however, that is not associated with a particular switch. It is the Overload indicator, located between the K1 and K2 electrode sections. This LED lights up whenever one of the amplifiers in the AFCBP1 is nearing saturation (i.e., an output greater than about 10.0 volts). Measurements made during an overload condition *are likely to be inaccurate!*

General Controls

POWER   The power switch is located in the lower left corner of the panel.

CONTROL SOURCE   A push button switch *with a one second delay* which switches control of the AFCBP1 between the front panel and an external computer. When control is passed to an external computer, all front panel controls are disabled and/or ignored, with the exception of the control source push button itself.

ELECTRODES   There are two push button switches that control cell connections to the bipotentiostat. The push button on the right (labelled "norm dummy") determines whether the circuitry of the bipotentiostat is connected to the external cell (normal) or to a set of internal resistors (dummy). The latter provides a convenient way to idle and/or test the instrument.

The push button on the left (labelled "open") is normally turned off when performing any type of voltammetry experiment. When turned on, the potentiostat measures the "open circuit potential" between the working electrode and the reference electrode. This essentially turns the bipotentiostat into a very expensive voltmeter for use with pH or other ion selective electrodes.

VOLTAGE   A knob type switch that determines which voltage appears on the voltmeter display. When in the "Sweep" position, the output of the Sweep Generator is displayed. When in the "E1" or "E2" position, the voltage presented at the "E1" or "E2" jack appears on the voltmeter display. When in the "I1" or "I2" position, the voltage presented at the "I1" or "I2" jack appears on the voltmeter display. This voltage reading may be converted to a current value by noting the setting of the appropriate current converter.

Sweep Generator Controls

ZERO A push button switch which sends the output of the Sweep Generator to zero volts. The indicator light associated with this switch is turned on and the output remains at zero volts until switch is pressed again.

SWEEP A push button switch which alternately halts and then restarts the sweep. Two indicator lights associated with this switch indicate whether the sweep is in a "hold" or "run" condition. The setting of this switch is overridden by the zero switch, described above.

MANUAL SWEEP Two momentary push buttons that are usually used to change the direction of a sweep in progress. When the left-hand "up" button is pressed, the voltage sweep proceeds in a positive direction. When the right-hand "down" button is pressed, the sweep proceeds in a negative direction. Sweep direction can be adjusted before or during a sweep, and two indicator lights above these push buttons indicate the present direction of the sweep. These two push buttons can also be used to adjust the Sweep Generator output when it is in a "hold" condition (see sweep control, above).

SWEEP RATE A four digit push-button potentiometer is used in conjunction with a push button switch to adjust the sweep rate. The push button is labelled "range", and it toggles between the two available sweep ranges as indicated by the associated indicator light. When the "<100 mV/sec" range is selected, the push-button potentiometer provides reproducible sweep rate settings from 0.00 to 99.99 mV/sec in 0.01mV/sec increments. When the "<1000 mV/sec" range is selected, the push-button potentiometer provides reproducible sweep rate settings from 0.0 to 999.9 mV/sec in 0.1mV/sec increments.

UPPER LIMIT The four digit push-button potentiometer is used in conjunction a push button switch below it to adjust the upper voltage limit for the sweep. The push button controls the sign ("+" or "−") of the upper limit voltage, and the push-button potentiometer provides reproducible voltage settings from 0000 to 9999 mV in 1 mV increments. An indicator light above the push button switch indicates the sign of the limit potential.

LOWER LIMIT The four digit push-button potentiometer is used in conjunction a push button switch below it to adjust the lower voltage limit for the sweep. The push button controls the sign ("+" or "−") of the lower limit voltage, and the push-button potentiometer provides reproducible voltage settings from 0000 to 9999 mV in 1 mV increments. An indicator light above the push button switch indicates the sign of the limit potential.

Note: The limits can be set both positive, both negative, or one positive and one negative; however, the upper limit must always be more positive than the lower limit., and the absolute difference between them should exceed ten millivolts.

STOP@LIMIT In general, when one of these controls is active, the sweep halts when it reaches the corresponding limit potential. More specifically, if the upper "STOP@LIMIT" is activated, a positive going sweep continues until it reaches the upper limit, while a negative going sweep halts immediately. Conversely, if the lower "STOP@LIMIT" is activated, a negative going sweep continues until it reaches the lower limit, but a positive going sweep will halts immediately. Activating both "STOP @ LIMIT" buttons halts the sweep immediately.

Electrode Controls

Both working electrodes (K1 and K2) operate independently of each other, and each has a set of independent, yet virtually identical, controls on the front panel. The only difference is that the K1 electrode can be operated in either "galvanostat" or "potentiostat" mode, depending on whether the K1 current or voltage is controlled, respectively.

SWEEP VOLTAGE When this push button switch is activated the light above it turns "on", and the output of the Sweep Generator is applied to the summing circuit of the working electrode. When turned "off", there is no internal connection between the Sweep Generator and the working electrode circuit.

OFFSET VOLTAGE This four digit push-button potentiometer is used together with two push buttons adjust an offset voltage applied to the summing circuit for the working electrode. The two push buttons control the sign ("+" or "−") of the offset voltage, and the push-button potentiometer provides reproducible voltage settings from 0000 to 9999 mV in 1 mV increments. If both push button switches are "off", then no offset voltage is applied to the working electrode.

CURRENT CONVERTER Two push button switches control the conversion factor used for scaling the working electrode current to a proportional voltage presented on the front panel. Seven decades of conversion factors are available from 100 mA/V to 100 nA/V.

MODE This push button is located in the K1 electrode section of the front panel, and it only applies to the K1 working electrode circuit. Pressing this button toggles the K1 electrode between the "galvanostat" mode and the "potentiostat" mode. The K2 electrode is always in "potentiostat" mode.

Experimental Voltammetry

The AFCBP1 may be used in a wide variety of electrochemical experiments, and some of the more popular electroanalytical methods are described in this section. Before using the instrument in any new application, be certain to consult the specifications for the AFCBP1 if there is any question that your experiment will exceed the capabilities of the instrument. Some general guidelines to keep in mind are discussed below.

The bipotentiostat circuit inside the AFCBP1 is designed for use in the "middle range" of currents usually encountered in electrochemical experiments. On the upper end, the maximum current that can be measured is one ampere, while on the lower end, the minimum current that can be measured is about one nanoamp. At the low current end, extreme care must be taken to shield the experiment from electrical noise, and a very sensitive recording device should be used.

The circuit inside the AFCBP1 is also designed for use in low to moderate frequency experiments. The operational amplifiers that make up the bipotentiostat's "feedback" circuit have a rise time of 0.5 volts per second, and the frequency response of the instrument, measured as the "small signal bandwidth", is at least 10 kHz. Results obtained from experiments using sweep rates exceeding ten volts per second or pulse widths shorter than ten microseconds should be viewed with caution.

When making connections to the AFCBP1, be careful not to short output signals to each other or to the DC common. All output signals are current limited to prevent damage to the potentiostat in the event of a short duration short circuit to DC common.

The AFCBP1 has a compliance voltage of ±10 volts, although most typical electrochemical measurements require only a ±2 volt window. On occasion, an experimental situation will result in a very high current or voltage condition which "saturates" one of the internal operational amplifiers. Whenever one of the amplifiers nears saturation, an the OVERLOAD indicator lights up to let you know that there could be an error in the measurement being made. The circuits continue to function even though the indicator is on.

The AFCBP1 can be used with two, three, or four electrodes. To use the AFCBP1 as a conventional three-electrode potentiostat or galvanostat, it is not necessary to make any connections to the K2 electrode circuit. To use the AFCBP1 for 2-electrode current vs. voltage measurements, use the K1 and REF electrode inputs, but also short the CE input to the REF input.

General Experimental Apparatus

All electrochemical experiments require roughly the same materials and apparatus. After choosing the AFCBP1 bipotentiostat, the next priority is choice of a data recording device. Common choices include an XY Recorder or a digital oscilloscope. Dual electrode experiments generally require a two pen recorder or a dual trace oscilloscope.

Additional instrumentation is required for certain applications. Hydrodynamic methods require an electrode rotator, such as the Pine AFASR Rotator. Potential step methods, such as chronoamperometry, require a programmable pulse generator.

Choice of working and reference electrodes is highly dependent on the particular electrochemical system being studied. Common working electrode materials include glassy carbon (GC), platinum (Pt), and gold (Au). Popular reference electrodes include the saturated calomel electrode (SCE) and the silver-silver chloride reference. The counter electrode, also called an auxiliary electrode, is typically a piece of platinum wire, but more elaborate designs are available.

Glassware used for electrochemical cells varies widely from one application to another. In general the cell must have provisions for mounting three (or four) electrodes, and for hydrodynamic experiments, the cell must have an opening suitable for the shaft of a rotating working electrode.

If dissolved oxygen is likely to interfere with an electrochemical measurement, the cell design should include appropriate connections for bubbling and blanketing the test solution with an inert gas. A cylinder of compressed nitrogen or argon, a gas regulator, and associated plumbing will also be required in these situations.

Electrolyte solutions suitable for use with electrochemical techniques are somewhat limited. Aqueous electrolyte solutions are easiest to prepare and are quite popular. An inert salt, such as potassium nitrate, dissolved in HPLC grade water works very well. Non-aqueous systems, however, can dissolve a wider range of compounds, including organic and organometallic substances. The most popular non-aqueous solvent systems are based on acetonitrile solutions of tetraalkylammonium salts.

Controlled Potential Techniques

Most popular electroanalytical techniques require a *potentiostat*, that is, a device capable of measuring the working electrode current while controlling its potential. These popular techniques include *cyclic voltammetry*, *chronoamperometry*, most *rotated ring-disk methods*, and *square wave voltammetry*, to name just a few. All of these techniques share the common trait that it is the working electrode potential that is the "controlled" variable in the experiment, while the working

electrode current is the "measured" variable. Because the AFCBP1 is a *bi*potentiostat, it can control two working electrodes in this fashion, and the potential applied to one working electrode does not affect the potential or the current at the other electrode.

Potential Sweep Methods

Perhaps the most popular of electroanalytical techniques, both *linear sweep voltammetry* (LSV) and *cyclic voltammetry* (CV) produce a quick survey of the electrochemical behavior of a system. Both methods are three electrode potentiometric methods, meaning that a potentiostat controls the potential of a working electrode (versus a reference) while simultaneously measuring the current at the working electrode. Current is essentially prevented from passing through the reference electrode by the use of a counter electrode.

In Linear Sweep Voltammetry, the potential at the working electrode is swept from an initial potential (E_1) to a final potential (E_2) at some predetermined sweep rate (v). Usually E_1 and E_2 are chosen to span a region of electrochemical interest, such as the formal potential, $E°$, of an electroactive analyte. Experimental results are typically presented as a plot of current versus applied potential, known as a *linear sweep voltammogram*.

Cyclic Voltammetry is an extension of Linear Sweep Voltammetry that uses a triangle wave rather than a simple potential ramp. The potential at the working electrode is swept at a predetermined rate (v) from an initial potential (E_1) to a another potential (E_2). Upon reaching this "potential limit", however, the direction of the potential sweep is reversed. The potential is swept back to a second potential limit (E_3). At this point, one "cycle" of the experiment is complete, but in many cases, it is desirable to cycle the potential several more times between E_3 and E_2. Frequently, the initial potential, E_1, coincides with one of the potential limits, E_2 or E_3, but this is not a requirement. Experimental results are typically presented as a plot of current versus applied potential, known as a *cyclic voltammogram*.

The Model AFCBP1 bipotentiostat is capable of performing either LSV or CV experiments through the use of its internal Sweep Generator. Because the output of the Sweep Generator unit can be applied to either electrode, it does not matter whether K1 or K2 is used as the working electrode. If desired, an external signal generator may be used to provide the voltage sweep. Figure 11 shows the connections required to configure the AFCBP1 for cyclic voltammetry using a single pen XY Recorder. A digital oscilloscope may also be used.

AFCBP1 Bipotentiostat

XY Recorder   Cell
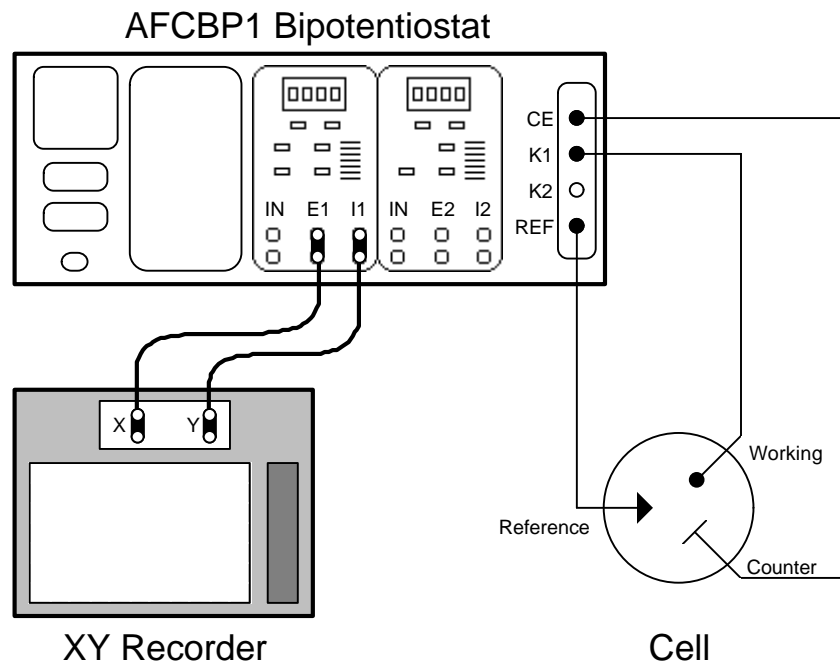
FIGURE 11:   CABLE CONNECTIONS FOR CYCLIC VOLTAMMETRY

Experiment (Cyclic Voltammetry)

This procedure illustrates how the AFCBP1 is used in a typical cyclic voltammetry experiment. Details of the procedure will vary according to the particular apparatus actually used.

Apparatus
1)   Pine Model AFCBP1 Bi-Potentiostat
2)   X-Y Recorder
3)   Electrochemical cell
4)   Platinum working electrode, Platinum counter electrode
5)   Saturated Calomel Electrode (reference)
6)   Cylinder of compressed, purified nitrogen for deaeration of solutions
7)   Various electrical cables with BNC, banana and alligator connectors

Reagents
5.0 m$\underline{M}$ potassium ferricyanide in 1.0 $\underline{M}$ potassium nitrate aqueous solution.

Procedure

NOTE: It is usually desirable to place the AFCBP1 in "Dummy" mode and lift the XY Recorder pen while an experiment is being set up. This prevents needless marks on the recorder paper.

1)   Assemble the electrochemical cell and fill it with the potassium ferricyanide solution. Mount the three electrodes in the cell. Connect the platinum working electrode to the K1 input. Connect the platinum counter electrode to the CE input. Connect the SCE reference to the REF input. The AFCBP1 should be in "Dummy" mode while electrode connections are being made. Bubble nitrogen through the ferricyanide solution for about 10 minutes to displace dissolved oxygen from the solution. You may perform steps 2 through 7 while bubbling.

2)   Connect the E1 output of the AFCBP1 to the X-axis input on the XY Recorder. Connect the I1 output of the AFCBP1 to the Y-axis input on the XY Recorder. Suggested settings for the recorder are 100mV/cm sensitivity on both axes and location of the origin near the center of the recorder paper.

3)   Set the K1 electrode controls as follows: Turn off the OFFSET VOLTAGE. Turn on the SWEEP VOLTAGE. Set the CURRENT CONVERTER to one of the lower sensitivities (such as 10mA/V) to prevent accidental overload while setting up.

4)   Set the voltmeter so that it monitors "E1", the working electrode (K1) potential. Make certain that K1 is in the "potentiostat" mode.

Note: Steps 5 & 6 (below) set up the AFCBP1 Sweep Generator to sweep the working electrode potential between +0.800 Volts and -0.200 Volts starting at an initial potential of +0.800 Volts. This range includes the formal potential of the ferricyanide/ferrocyanide redox couple.

5)   Set up the Sweep Generator as follows: Set the UPPER LIMIT to +0800 mV. Set the LOWER LIMIT to -0200 mV. Set the SWEEP RATE at 100.0 mV/sec.

6)   This step illustrates how to make the initial potential the same as a limit potential.
    a)   Activate the ZERO push button. The voltmeter display should read "0.00".
    b)   Push the MANUAL SWEEP button that is labelled with an "up" arrow.
    c)   Next, press the SWEEP button so that it indicates "Run" mode.
    d)   Activate the STOP @ LIMIT for the UPPER LIMIT.
    e)   Deactivate the ZERO push button. Observe the K1 potential ramp to 800mV.
    f)   Put the SWEEP control in the "hold" mode.
    g)   Deactivate the STOP @ LIMIT switch.

7)   Switch the potentiostat from "dummy" to "normal" and activate the XY plotter pen.

8)   To begin the experiment, put the SWEEP control in the "Run" position.

9)   A cyclic voltammogram should be traced on the XY Recorder. The Y-axis sensitivity will probably have to be adjusted so that the current signal expands to fill the recorder paper. The CURRENT CONVERTER setting may be adjusted to achieve the desired effect.

Notes

There is an alternate way to perform steps 5&6 that uses the Sweep Generator in conjunction with the OFFSET VOLTAGE to produce the desired potential sweep. The K1 OFFSET VOLTAGE is set to the initial potential, +0800 mV, while the Sweep Generator UPPER LIMIT is set to 0000 mV, and the LOWER LIMIT is set to -1000 mV. Because the voltage applied to the working electrode is the sum of the offset voltage and the Sweep Generator output, these settings have the same effect as those in the main procedure above. The advantage to this variation is that moving to the initial potential is easily accomplished by zeroing the Sweep Generator output. The (minor) disadvantage is that the upper and lower sweep limits cannot be directly read off of the front panel of the instrument.

To obtain a Linear Sweep Voltammogram, use the STOP @ LIMIT switch to halt the experiment after the first sweep.

Potential Step Methods

Several electrochemical experiments involve the application of one or more voltage pulses to the working electrode. The most common of these is simple *chronoamperometry* (CA), but variations of this basic experiment, including *chronocoulometry* (CC) and *double potential step chronoamperometry* (DPSCA), are also popular. All of these methods are three electrode potentiometric methods, meaning that a potentiostat controls the potential of a working electrode (versus a reference) while simultaneously measuring the current at the working electrode. Current is essentially prevented from passing through the reference electrode by the use of a third electrode, known as either the counter electrode or the auxiliary electrode.

Chronoamperometry involves a single voltage pulse (or potential step). The working electrode is initially at a potential, E_1, and it is quickly stepped to another potential, E_2. The time, t, at which the potential step is applied is taken as t = 0. Usually E_1 and E_2 are chosen to span a region of electrochemical interest, such as the formal potential, E°, of an electroactive analyte. A current transient is observed in response to the sudden change in electrode potential as electrons are transferred between the working electrode and the analyte in the adjacent solution. In an unstirred solution, the rate of this transfer is limited by how fast the analyte can physically diffuse to the electrode surface. An equation for this so-called "diffusion controlled" current, *i(t)*, was developed in 1902 by Cottrell as follows:

$$i(t) \ = \ n \, F \, A \, D^{1/2} \, C \ (p \, t)^{-1/2}$$

where *n* is the number of electrons exchanged between an analyte and the electrode, *F* is Faraday's constant, *A* is the area of the electrode, *D* is the diffusion coefficient of the analyte, *C* is the molar concentration of the analyte in the solution, and *t* is time since application of the pulse. Experimental results are typically presented as a Cottrell plot, which is a plot of current versus the inverse square root of time (*i versus t* $^{-1/2}$). The slope of such a plot yields the quantity $D^{1/2} \, C$, and in most cases, the value of either *D* or *C* is known from previous experiments.

While the AFCBP1 has no built in circuitry for generating voltage pulses, it is possible to connect a third-party pulse generator to a potential input on the AFCBP1. With the instrument set in "potentiostat" mode, pulses from the external pulse generator are applied to the working electrode, and the current at this electrode is presented by the AFCBP1 for recording by either a YT Recorder or digital oscilloscope. If the only recorder available is an XY Recorder, the Sweep Generator on the AFCBP1 can be made to function as a time base for the X-axis on the recorder. Figure 12 illustrates how the AFCBP1, a pulse generator, and a digital oscilloscope can be used for chronoamperometry.

AFCBP1 Bipotentiostat
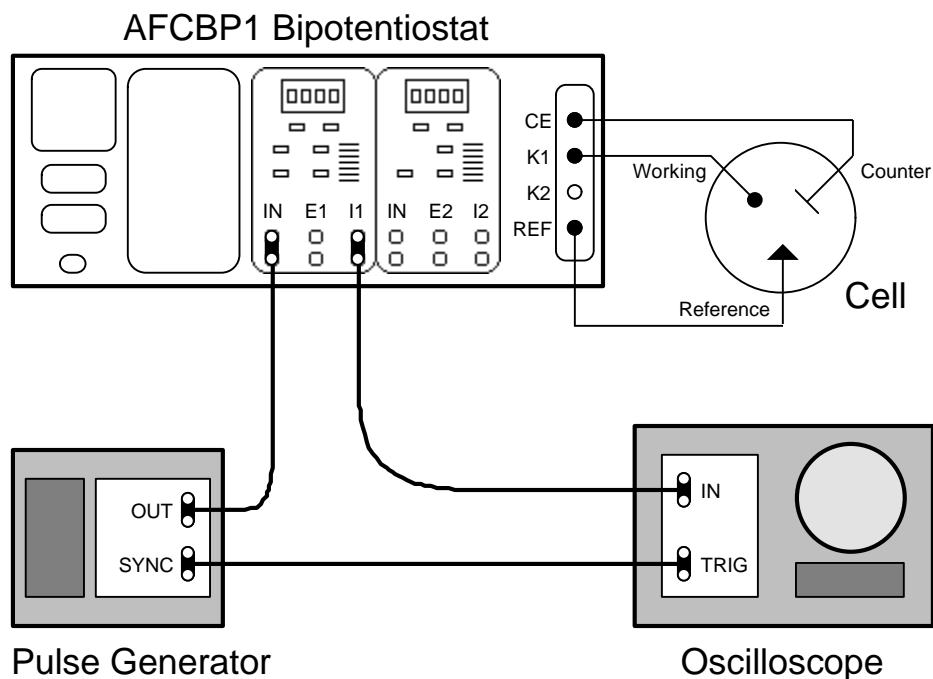
FIGURE 12:   **CABLE CONNECTIONS FOR CHRONOAMPEROMETRY**

Hydrodynamic Methods

Electrochemical experiments where the solution is moving with respect to the electrode (or vice versa) are termed hydrodynamic experiments. The most popular of these are the forced convection methods involving either a *rotating disk electrode* (RDE) or a *rotating ring-disk electrode* (RRDE). The rotating motion of the electrode sets up an easily characterized solution flow within the electrochemical cell. Using the electrode surface as a frame of reference, the solution immediately adjacent to the surface appears stagnant while the remaining bulk of the solution is being stirred.

Analyte from the bulk of the solution is brought near to the electrode by the convective (stirring) action of the electrode. Once near to the electrode, physical diffusion is responsible for bringing the analyte to the electrode surface. This second process determines how fast analyte reaches the electrode surface, and this rate of diffusion will be reflected in the observed current at the electrode. Of course, current will only be observed when the electrode potential is suitably poised to either oxidize or reduce the analyte once it arrives at the electrode surface.

The AFCBP1 bipotentiostat was designed for use in both RDE and RRDE voltammetry. In both experiments, the potential of the disk electrode is typically scanned through a region of electrochemical interest using the Sweep Generator, and the disk current is plotted versus the disk potential. A steady state voltammogram with a sigmoidal shape usually results. In RRDE, the current at the ring electrode is also monitored, and a dual pen recorder is required. Figure 13 depicts typical cable connections required for an RRDE

experiment. To set up an RDE experiment, simply omit all connections associated with the K2 electrode.
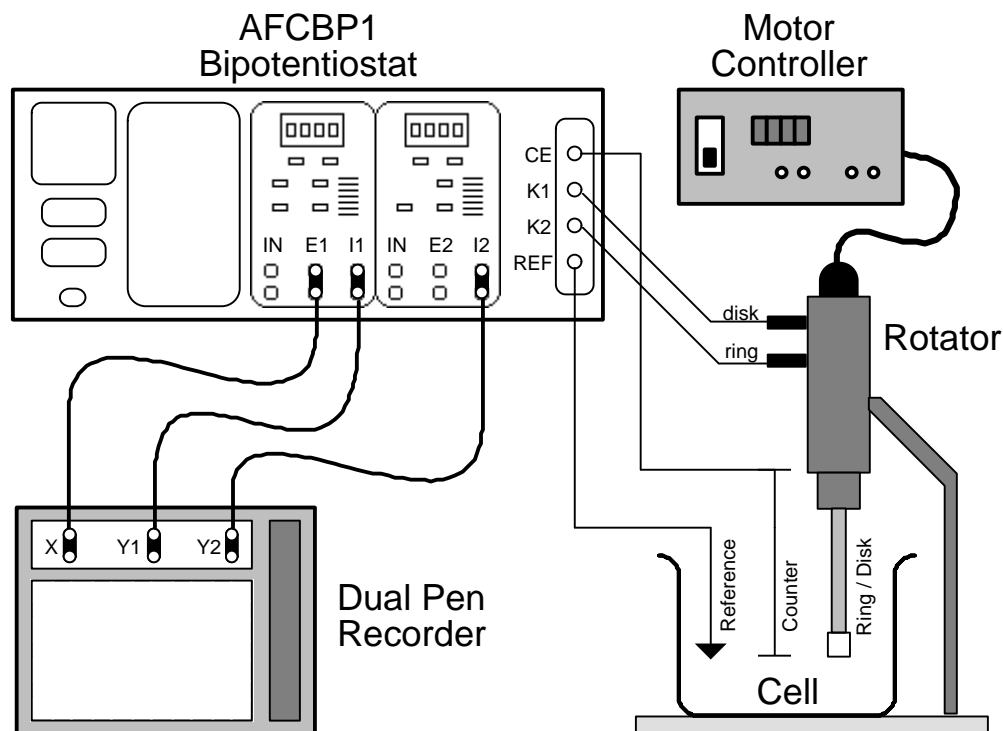
FIGURE 13: CABLE CONNECTIONS FOR RRDE VOLTAMMETRY

Controlled Current Methods

Electroanalytical methods in which the working electrode current is the controlled variable, and the working electrode potential is the measured variable, require the use of a *galvanostat* rather than a potentiostat. These methods are usually referred to as *chronopotentiometric* methods and involve the use of three electrodes. The galvanostat passes a controlled current between the working and counter electrodes while at the same time monitoring the potential of the working electrode with respect to a reference electrode. Chronopotentiometric methods are classified according to the way in which the current is controlled, and the simplest is *constant current chronopotentiometry*.

When the MODE push button (in the K1 electrode section) is switched to the "Galvanostat" position, the operation of working electrode K1 is switched from potential control to current control. As always, the potential at the K1 electrode may be monitored at output "E1", and the current at the K1 electrode may be monitored (as a proportional potential) at the "I1" output. The setting of the CURRENT CONVERTER plays its usual role in determining the proportionality constant for calculating the K1 current from the voltage presented at the "I1" output.

The K1 CURRENT CONVERTER setting also plays a second role when operating in galvanostatic mode. Because the controlled variable is the working

electrode current, it is necessary to have some way of specifying just how much current is to be passed through the working electrode. This may be accomplished by using a *voltage* signal from the OFFSET VOLTAGE, the SWEEP GENERATOR output, an external signal applied to the "IN" input, or any combination of the three. The sum of all three of these voltages is converted (internally) to a current according to the CURRENT CONVERTER setting. It is this resulting current that is then applied to the K1 working electrode.

The types of controlled current experiments that can be performed include (1) constant current chronopotentiometry, (2) current ramp chronopotentiometry, (3) current reversal chronopotentiometry, (4) cyclic chronopotentiometry, and (5) so-called "diffusion layer" titrations using a rotated ring-disk electrode (RRDE). To perform a chronopotentiometric experiment, the AFCBP1 should be configured as shown in Figure 12, except that the "E1" output should be connected to the recording device. The pulse generator may or may not be required.

Constant Current Chronopotentiometry

In this experiment, the current is simply stepped from zero to some value which is held constant for the duration of the experiment. As the analyte is either oxidized or reduced at the electrode surface, the potential of the electrode changes according to the ratio of oxidized to reduced analytes as described by the Nernst equation. Typically, results are plotted as working electrode potential versus time. Such a plot is called a *chronopotentiogram* and typically exhibits a sigmoidal shaped curve. The start of the curve is at time zero, and the sigmoid ends at the so-called transition time, t_T. The Sand equation (below) relates the transition time to the current, *i*, and analyte concentration, *C*.

$$i \,=\, 0.5\,n\,F\,A\,D^{1/2}\,p^{1/2}\,C\ t_T{}^{-1/2}$$

where *n* is the number of electrons exchanged between an analyte and the electrode, *F* is Faraday's constant, *A* is the area of the electrode, *D* is the diffusion coefficient of the analyte, *C* is the molar concentration of the analyte in the solution.

To produce the sudden current step for *constant current chronopotentiometry*, use the K1 OFFSET VOLTAGE. The OFFSET VOLTAGE should initially be turned off by deactivating both the "+" and the "-" push button switches (indicator lights off). The magnitude of the desired current step may be set using the four digit push-button potentiometer, noting that this magnitude is scaled by the setting of the CURRENT CONVERTER. The current step can then be applied suddenly at any time simply by pressing either the "+" or the "-" push button. In a typical experiment the potential response would be monitored as a function of time from the moment the current step is applied.

Current Ramp Chronopotentiometry

To produce a smooth analog current sweep, use the SWEEP GENERATOR output. The SWEEP GENERATOR should be set up to provide a voltage sweep that begins at zero volts and ends at some potential corresponding to whatever final current is desired. As always, the proportionality between this final potential and the final current is controlled by the setting of the CURRENT CONVERTER. Similarly, this setting governs the relationship between the rate of the potential sweep and the rate of the current sweep. In a typical experiment the potential response would be monitored as a function of time from the moment the sweep commences.

Other Galvanostatic Methods

Both *current reversal chronopotentiometry* and *cyclic chronopotentiometry* require that the current be controlled by a pulse sequence. While the AFCBP1 has no internal circuitry that allows pulse programming, it is possible to connect a third-party pulse generator to the "IN" input for the K1 electrode. The incoming signal should be set up to provide a voltage pulse corresponding to whatever current pulse sequence is required. The proportionality between then incoming voltages and the applied currents is controlled by the setting of the CURRENT CONVERTER. In a typical experiment the potential response would be monitored as a function of time from the moment the pulse sequence commences.

Galvanostatic control of the disk electrode of a RRDE, with potentiostatic control of the ring electrode, can be applied for quantitative analytical determinations of electroinactive species, by way of so-called "diffusion layer titrations", and for the measurement of rate constants for pseudo second-order homogeneous reactions.

Open Loop Potentials

The AFCBP1 can be used to make simple potentiometric measurements by placing it in "open loop potential" mode. When in this mode, the function of the bipotentiostat is essentially changed to that of a high input impedance digital voltmeter. The potential difference between a working electrode (K1 and/or K2) and the reference electrode is measured at zero current, and this potential is presented at the appropriate front panel output jack (E1 and/or E2). The built-in voltmeter may be used to monitor these potentials, but it may lack the precision required in certain applications.

Most often, the "open loop" feature is used in conjunction with a pH or ion selective electrode together with a reference electrode, such as a standard calomel electrode (SCE). The pH or ion selective electrode is connected to the K1 input jack, and the reference electrode is connected to the REF input jack. It is actually possible to have two pH or ion selective electrodes operating simultaneously by connecting one of the electrodes to K1 and the other to K2. Note that only the (center) signal line on the REF BNC connector should be

connected to the reference electrode. The counter electrode (CE) is not used for open loop measurements.

To put the AFCBP1 in "open loop" mode, use the two front panel push buttons shown in Figure 14. The left hand button is used to turn on "open loop" mode, and the right hand button is used to select "normal" mode. (If the bipotentiostat is put in "dummy" mode, the open loop potential goes to zero volts.)
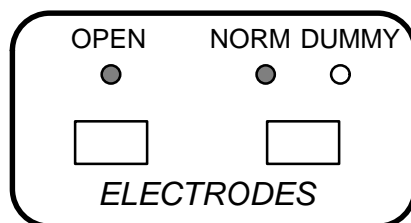
FIGURE 14:   "OPEN LOOP" SETTINGS

Some pH and ion selective electrodes are equipped with an internal reference electrode. This type of electrode will have two separate leads coming out of it, although many times this fact is disguised by the use of two conductor coaxial cable. When using this type of electrode, it is important to determine which lead connects to the internal reference electrode and which connects to the sensing electrode.
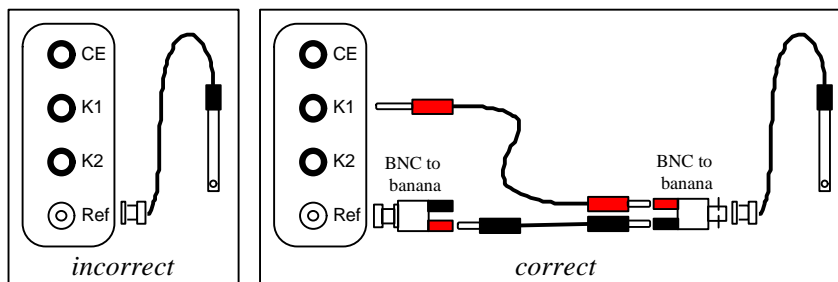
FIGURE 15:   PROPER pH ELECTRODE CONNECTIONS

For example, a typical "combination" pH electrode shown in Figure 15 has a single coaxial cable coming out of it which terminates with a standard BNC connector. The inner (signal line) part of the BNC connector makes contact with the sensing electrode, while the outer (shield) part makes contact with the internal reference electrode. It is incorrect to directly connect the pH electrode to the REF jack on the AFCBP1; rather, a pair of BNC-to-banana adapters and two jumper cables are used to correctly connect the pH electrode to the bipotentiostat.

Figure 16 shows measurements of open loop potential made with a pH electrode immersed in three different buffer solutions. A linear relationship between potential and pH is observed, as expected, with the slope of the calibration curve being close to the ideal value of 59 mV/pH unit.

Open Loop Potential of a pH Electrode

(Fisher Catalog # 13-620-108)

slope = 56.59 mV/pH unit
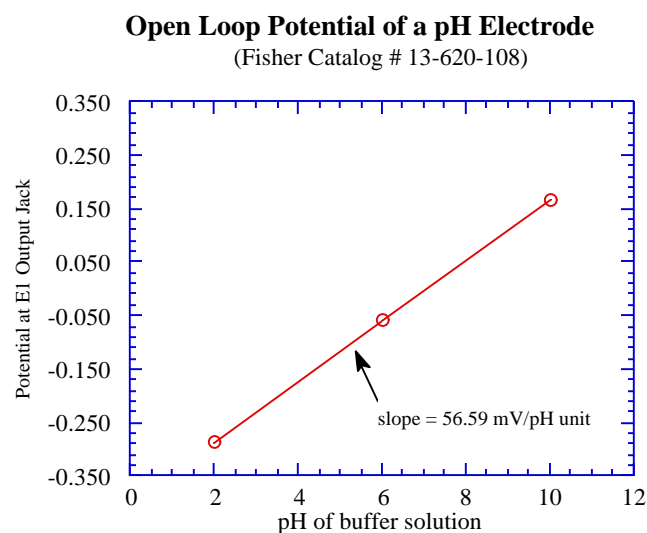
FIGURE 16:   VOLTAGE RESPONSE OF A PH ELECTRODE IN VARIOUS BUFFERS

Four Electrode Arrangements

Bipotentiostats have found use in several types of four electrode experiments other than the traditional rotated ring-disk variety. Many flow cell designs call for potential control at two working electrodes. This ability to independently control two working electrodes has been used successfully to generate concentration gradients across thin layers of liquid solutions and conducting polymer films. Figure 17 depicts three examples of electrode arrangements which require a bipotentiostat.

A common feature shared by all three cells depicted in Figure 17 is that the reference electrode is located close to the K1 working electrode. While the K1 and K2 circuitry do operate independently and are equivalent in function, the possibility exists for the two electrodes to interact via the uncompensated solution IR drop in a particular electrochemical cell. Error from this interaction is minimized by locating the reference electrode close to the K1 working electrode. Some attention to this matter is required when designing any four electrode arrangement.

The **interdigitized array electrode (IDA)** is usually fabricated on a silicon substrate as a microlithographically defined array of parallel microband gold or platinum electrodes. Alternating microbands are in electrical contact, and the region of experimental interest is the gap between each alternate finger, which is typically from 1 to 50 micrometers across. It is across this small gap that a classic "collection" experiment can be performed. An electroactive analyte may be oxidized at the electrode on one side of the gap, allowed to diffuse across the gap, and then reduced at the opposing electrode. These experiments may be conducted in traditional electrolyte solutions or with polymeric electrolyte solvents, such doped poly(ethylene oxide).

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see: C.E.D. Chidsey, B.J. Feldman, C. Lundgren, R.W. Murray, *Anal. Chem. 58* (**1986**) 601.
          K. Aoki, M. Morita, O. Niwa and H. Tabei, *J. Electroanal. Chem. Soc. 106* (**1984**) 7389.

The **polymer sandwich electrode** arrangement is frequently used to study a special type of conducting polymer called a "redox conducting polymer". Of interest is measuring the mobility of electrons as they "hop" through the thin layer of polymer from one working electrode to the other. Usually, the sandwich is immersed in electrolyte solution along with a reference and a counter electrode. Because of the relatively high resistance of the polymeric film, it is important that the inner (platinum) electrode not be connected to the K1 input. Rather, the outer (gold) electrode should be connected to the K1 input because it is closer to the reference electrode.

see: C.E.D. Chidsey and R.W. Murray, *Science 231* (**1986**) 25.
          P.G. Pickup and R.W. Murray, *J. Am. Chem. Soc. 105* (**1983**) 4510.

Another cell requiring the use of four electrodes is the **dual electrode thin layer cell**. Two opposing working electrodes, separated by a small gap, are used to generate a concentration gradient across the gap. Typically, one electrode is held at a constant potential while the other is scanned past the formal potential of the redox couple being studied. When the potential difference across the gap becomes large enough, analyte is oxidized at one electrode and reduced at the opposing electrode. A steady-state current response similar to that of a rotated disk electrode is observed.

see: L.B. Anderson and C.N. Reilly, *J. Electroanal. Chem. 10* (**1965**) 295, 538.
          G.P. Kittlesen, H.S. White, and M.S. Wrighton, *J. Am. Chem. Soc. 106* (**1984**) 7389.

Interdigitized Array Electrode

Conducting Polymer Film "Sandwich"

Dual Electrode Thin Layer Cell
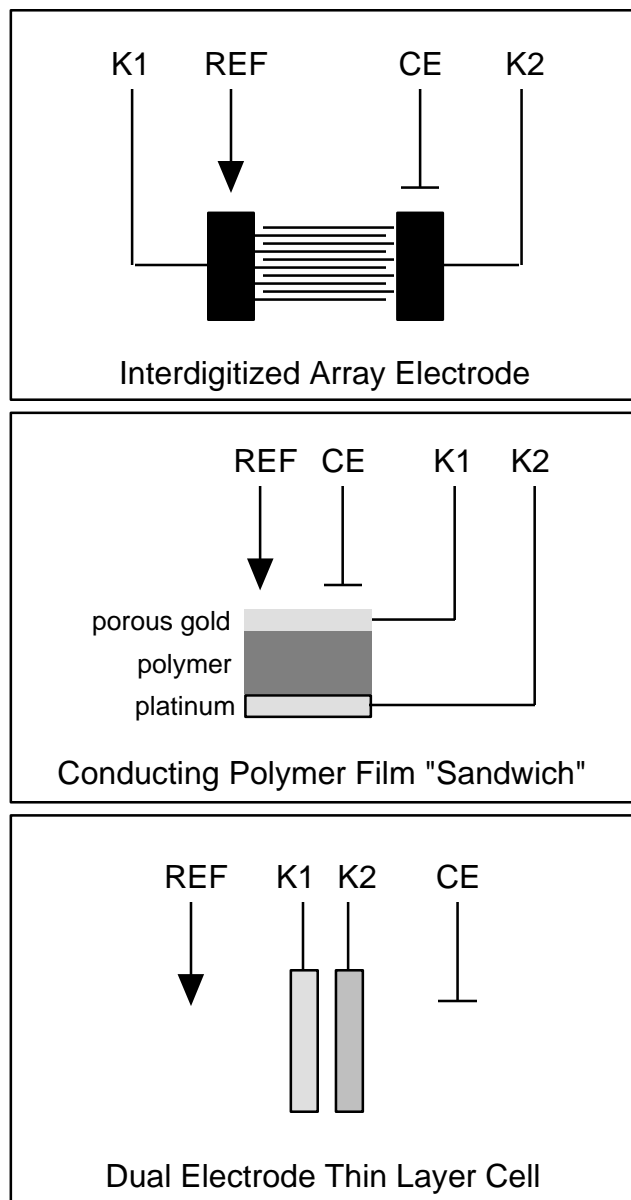
FIGURE 17: TYPICAL FOUR-ELECTRODE ARRANGEMENTS

Two Electrode Arrangements

The AFCBP1 can be used to trace the current vs. voltage response across just two electrodes in an electrochemical cell. This usually results in a measurement of cell conductivity.

In order to use the AFCBP1 in this fashion, one of the cell's electrodes is connected to the K1 jack on the AFCBP1, and the other is connected to the CE jack. Also, the REF and CE inputs are shorted together to prevent overload. (The outer shield on the REF jack is unused).

The Sweep Generator output, the K1 Offset Voltage, and/or any external voltage source are summed and applied as a potential difference across the two electrodes. The current through the cell is presented at the "I1" jack as a proportional voltage according to the setting of the CURRENT CONVERTER.

Proper Grounding

The electrochemical cell should be placed in a "Faraday cage" to shield it from laboratory electrical noise. A Faraday cage is simply a small cabinet which is covered with some form of shielding, such as a metal sheet or screen. Typically, this shielding is grounded to a DC Common jack on the AFCBP1. The eight black banana jacks on the front panel provide easy access to the AFCBP1's DC Common.

Recording devices, including XY Recorders and digital oscilloscopes should also be grounded to a DC Common jack. Other items associated with an electrochemical experiment may require grounding. These include the following: support racks, push carts, glove boxes, stir plates, and nearby computers.

The AFCBP1's DC Common floats with respect to the instrument's chassis. In some cases it may be necessary to connect one of the black banana jacks to earth ground in order to reduce noise. A binding post (banana type) connected to the instrument's chassis is provided on the front panel for convenience in making this connection. The instrument chassis is also connected to the normal ground pin on the 115 Volt 3-prong plug. If the instrument is plugged into an outlet with a good quality earth ground, then the front panel binding post will be at earth ground as well.

All cables connected to the AFCBP1 front panel, especially the electrode connections, should be kept as short as possible. Because the REF electrode jack presents a very high input impedance, it is susceptible to picking up unwanted noise. The AFCBP1 incorporates an insulated BNC type connector for the REF electrode connection, allowing complete shielding of the REF input lead. The shield (outer portion of the BNC connector) is driven to the same voltage as the REF signal, and should not be grounded. In addition, the shield should not

be connected to any other point at the electrode end, so that no current flows in the shield.

It is suggested that no unusual connections be made to the ELECTRODE jacks as they are intended to connect only to the actual electrodes. The REF jack is particularly sensitive to such connections. It is permissible, however, for the CE and REF jacks to be shorted together when the potentiostat is being used for two electrode measurements.

Keep in mind that some noise is inherent to all measurement systems, although it may not always be apparent. Traditionally, XY plotters have been used extensively to record the results of electrochemical experiments. These plotters usually have an internal RC circuit that limits the frequency response of the plotter, thus filtering out most noise. When a computerized data acquisition system is used, however, there is usually no such filter between the potentiostat and the computer. This means that "raw" data acquired by the computer may appear (shockingly) noisy, especially to those whose only past experience has been with XY plotters. Most electrochemical software, including that offered by Pine Instrument Company, will provide the user with a range of data smoothing options to help eliminate noise.

Oscillations

The AFCBP1 is an analog system which relies on feedback for stable operation. There is the possibility, on certain systems, that the feedback may become phase shifted and cause the unit to oscillate. Symptoms of oscillation include unexplained jumps in potential or current, or when the overload indicator is glowing for no apparent reason. The presence or absence of oscillation is verified by using an oscilloscope to observe the output voltages.

If oscillations are observed, and the grounding techniques discussed above do not eliminate the problem, then you should try slowing the response time of the AFCBP1. Located on the back panel are six jacks to which capacitance may be connected. It is suggested that capacitors in the range of 0.001 to 0.1 microfarads be added across the K1 and/or K2 jacks on the back panel. Various combinations should be tried, as experience will best dictate what should be used. The capacitors should be either film or ceramic type and rated for at least 30 volts.

If problems are experienced in the galvanostatic mode of operation, there are two jacks on the rear panel for external compensation in that mode only. Capacitors in the range of 0.001 to 0.1 microfarads are recommended.

Troubleshooting

When a malfunction or some anomalous behavior is observed during an electrochemical experiment, one must first determine whether the difficulty originates within the potentiostat or is external to the potentiostat. In most cases, proper operation of the AFCBP1 potentiostat can be verified without making any external connections. The following procedures allow general testing using an internal "Dummy" cell resistor network rather than an actual electrochemical cell. These procedures do assume that the internal voltmeter is working properly, but if there is reason to believe that the voltmeter is malfunctioning, an external voltmeter can be used in its place. Testing procedures should be followed in the order they are presented.

Testing the Sweep Generator

1)   Turn on the AFCBP1 bipotentiostat. Several small red indicator lights should light up indicating that the power is on.

2)   Set the CONTROL SOURCE to "panel" mode (keep in mind that this control has a one second delay time). Set the NORM vs DUMMY control to "dummy" mode. Make certain that the OPEN control is deactivated. Rotate the VOLTMETER knob to the "sweep" position.

3)   Set the upper limit to +9000mV, and the lower limit to -9000mV. Set the SWEEP RATE to 500.0 mV/sec using the "<1000 mV/Sec" setting on the RANGE control. Deactivate both STOP @ LIMIT controls. Activate the ZERO control. Put the SWEEP control in the "hold" position.

4)   Deactivate the SWEEP VOLTAGE switches for both working electrodes. Turn off the OFFSET VOLTAGE controls for both working electrodes. Set the CURRENT CONVERTER controls for both working electrodes to the "1 mA" position. Set the K1 electrode's mode control to the "pot" setting.

5)   At this point, confirm that the Voltmeter display reads "0.00" volts.

6)   Deactivate the ZERO control. Again, confirm that the Voltmeter display is "0.00" volts.

7)   Put the SWEEP control in the "run" position and observe the Voltmeter. The digit immediately to the *left* of the decimal point (*i.e.*, the ones place) should change by one digit every 2 seconds. Next, change the RANGE control to the "<100 mV/Sec" position and observe the Voltmeter again. Now, the digit immediately to the *right* of the decimal point (*i.e.*, the tenths place) should change by one digit every two seconds. Return the RANGE control to the "<1000 mV/Sec" position.

8)   Put the SWEEP control in the "hold" position and observe the Voltmeter. The value displayed on the Voltmeter should remain constant (the last digit may fluctuate ±1 digit). Activate the ZERO control. Confirm that the Voltmeter display reads "0.00" volts.

9)   Momentarily press the "down arrow" MANUAL SWEEP button to set the sweep direction to "down". Confirm that the Voltmeter display still reads "0.00" volts. Deactivate the ZERO control and again confirm that the Voltmeter still reads "0.00" volts. Put the SWEEP control in the "run" position and observe the Voltmeter. The reading on the

Voltmeter should begin to decrease (becoming more negative). Momentarily press the "up arrow" MANUAL SWEEP button and observe the Voltmeter. The reading on the Voltmeter should begin to increase (becoming more positive).

10) Activate the ZERO control. Momentarily press the "up arrow" MANUAL SWEEP button to set the sweep direction to "up". Activate the STOP @ LIMIT for the UPPER LIMIT. Deactivate the ZERO control. The Voltmeter reading should climb to "9.00" and stop. Next, activate the STOP @ LIMIT for the LOWER LIMIT. Then, deactivate STOP @ LIMIT for the UPPER LIMIT. The Voltmeter reading should move to "-9.00" and stop.

The ten steps above test for proper behavior of the major functions of the Sweep Generator. If no problems are encountered, the Sweep Generator is most likely functioning properly.

Testing an Electrode Unit (K1 or K2)

1) After testing for proper operation of the Sweep Generator (above), leave the AFCBP1 turned on, and follow these directions:

2) Set the CONTROL SOURCE to "panel" mode (keep in mind that this control has a one second delay time). Set the NORM vs DUMMY control to "dummy" mode. Make certain that the OPEN control is deactivated. Rotate the Voltmeter knob to the "E1" or "E2" position, depending on which electrode unit is being tested.

3) Set the UPPER LIMIT to +1000mV, and the LOWER LIMIT to -1000mV. Set the SWEEP RATE to 50.00 mV/sec using the "<100 mV/Sec" setting on the RANGE control. Deactivate both STOP @ LIMIT controls. Activate the ZERO control. Put the SWEEP control in the "hold" position.

4) Deactivate the SWEEP VOLTAGE switches for both working electrodes. Turn off the OFFSET VOLTAGE controls for both working electrodes. Set the CURRENT CONVERTER controls for both working electrodes to the "1 mA" position. Set the K1 electrode's MODE control to the "pot" setting.

5) At this point, confirm that the Voltmeter display is reads "0.00" volts.

6) Set the OFFSET VOLTAGE for the electrode being tested at -1000mV. The voltmeter should read "-1.00". Now, set the OFFSET VOLTAGE at +1000mV. The voltmeter should read "1.00" volts.

7) Rotate the Voltmeter knob to the "I1" or "I2" position, depending on which electrode is being tested. The voltmeter should read "1.00", but may deviate up to 1% from this value. Next, using the CURRENT CONVERTER control, check every other setting from "100 mA" down to "100 nA" to verify the other Voltmeter readings listed in the table:

CURRENT CONVERTER Setting	Expected Voltmeter Reading (I1 or I2)
100 mA	"0.01"
10 mA	"0.10"
1 mA	"1.00"
100 μA	"10.00" (with possible OVERLOAD)
10 μA	"13.50" or higher with OVERLOAD!
1 μA	"13.50" or higher with OVERLOAD!
100 nA	"13.50" or higher with OVERLOAD!

8)   Return the CURRENT CONVERTER control to the "1 mA" position. Deactivate the OFFSET VOLTAGE. Activate the SWEEP VOLTAGE control. Verify that the Voltmeter is still displaying the current ("I1" or "I2"), and that it reads "0.00".

9)   Deactivate the ZERO control. Initiate a voltage sweep by changing the setting of the SWEEP control to "run". Observe the voltmeter reading. It should slowly sweep from 0.00 up or down to 1.00 or -1.00 volts. (The initial sweep direction may be set "up" or "down" by toggling the MANUAL SWEEP control in the desired direction; otherwise, the initial direction is random). It should not matter whether the potential "E" or the current "I" is displayed on the Voltmeter. In either case, it should display a sweep between "1.00" and "-1.00" volts.

10)   **For electrode K1 only:** Change the K1 MODE control to "gal". It should not matter whether the potential "E1" or the current "I1" is displayed on the Voltmeter-in either case it should display a sweep between "1.00" and "-1.00" volts.

If no problems are encountered, the electrode unit is most likely functioning properly. The next section describes how to troubleshoot problems outside of the potentiostat itself.

Further Troubleshooting

If the testing procedures outlined in the previous section indicate that the AFCBP1 is functioning properly, then it is quite likely that there is a problem in the external electrochemical cell or connections. The suggestions below describe common problems encountered during electrochemical experiments. Common mistakes, made even by experienced electrochemists, are included as well. Symptoms are described for scanning experiments, such as cyclic voltammetry, but the suggestions apply to other methods as well.

Symptom:	**No observed current.** The XY Recorder shows no motion on the Y axis (current), but the X axis (voltage) is functioning properly. "I1" (or "I2") voltmeter reading remains at or near zero.
Suggestions:	**Low Current Sensitivity.** The CURRENT CONVERTER setting is too high for a signal to be detected by XY Recorder. The Y axis sensitivity on the recorder may also be too low. If the CURRENT CONVERTER is on the most sensitive setting ("100 nA") and there is still no observed current, then there is probably a discontinuity in the working electrode connection.
	Working Electrode Discontinuity. This is most often caused by the working electrode failing to contact the solution. This happens whenever the surface of the electrode is fouled or when there is a gas bubble trapped on the surface. Also, the cable connecting the working electrode to the AFCBP1 may have an open circuit. This frequently happens near jacks at the cable ends.

Symptom:	**No motion on X axis (voltage).**
Suggestion:	**Sweep is not being applied to working electrode.** The sweep is not turned on. The SWEEP VOLTAGE push button for the working electrode in question is deactivated. The XY Recorder may also be on "stand-by".

Symptom:	**Linear Current Response.** During cyclic voltammetry, the XY Recorder draws a smooth diagonal line. During step experiments, such as chronoamperometry, a horizontal line is observed instead of a decay transient.
Suggestion:	**Dummy Cell Electrochemistry.** The potentiostat is in "Dummy" mode. Put it in the "Normal" mode and try again.

Symptom:	**OVERLOAD Indicator ON.** Overload occurs whenever potentiostat is placed in "Normal" mode, regardless of applied potential.
Suggestions:	**Low Current Sensitivity.** The CURRENT CONVERTER is set to too sensitive a setting, and this is saturating an internal amplifier. **REF to CE Discontinuity.** During normal cell operation, there is a always an electrically conductive path between the REF and CE jacks. This path consists of cables running to the reference and counter electrodes, and is completed by both electrodes being immersed in the same conductive electrolyte solution. Overload occurs when this path is broken. Check cables to the reference and counter electrodes. If cables are okay, there could be a problem with the cell. Does the test solution contain enough electrolyte? Have any solutions dried up? Is a glass frit clogged? Is there a bubble in a Luggin capillary? Is the reference electrode defective? **Short Circuit between K1 and K2.** In dual working electrode experiments, a direct short between the two electrodes will result in an overload. Shorted ring-disk electrodes, interdigitized arrays, and thin layer cells will cause an overload.

Symptom:	**Noise.**
Suggestions:	**Rotating electrode experiments.** Dirty electrode contacts on rotator or electrode shaft. Clean gently with abrasive paper. Dirty rotator belt results in build up of a large electrostatic charge. Clean belt by soaking for 3 hours in nitric acid (50% or 6$\underline{M}$) at room temperature. Dispose of acid properly!
	Gas bubbles. Bubbles are impinging on electrode surface during experiment. The gas may be generated electrochemically or nitrogen deareation may have been left on accidently.
	Discontinuity. There may be an intermittant discontinuity in an electrode connection. Wiggle each lead separately to determine source.
	Poor Grounding. See earlier section on noise and also check for ground loops.
	Noise Pickup. When operating the AFCBP1 at a high current sensitivity, a Faraday cage will most likely be required. The reference electrode is very sensitive to external noise sources, and may require more elaborate shielding. Thermostated laboratory ovens, variacs, and hot plates generate considerable power line noise.

Technical Information

Bibliography

The following books are recommended for information about electrochemistry.

Kissenger, P.T. and Heineman, W.R. (eds): *Laboratory Techniques in Electroanalytical Chemistry* (Dekker, New York, 1984). This book presents a more practical side of electroanalytical chemistry, providing information about instrument design, choice of chemicals, and data analysis.

Bard, A.J. and Faulkner, L.R.: *Electrochemical Methods* (Wiley, New York, 1980). Widely considered to be the final authority in modern electroanalytical chemistry, this book presents highly detailed technical discourses on just about every conceivable routine electrochemical experiment. It is very often used as textbook for graduate level courses.

Sawyer, D.T., Heineman, W.R., and Beebe, J.M: *Chemistry Experiments for Instrumental Methods* (Wiley, New York, 1984). The first section of this undergraduate laboratory textbook describes a wide variety of electrochemical experiments suitable for use in college instruction. Proven experiments are presented with detailed instructions on how to conduct them.

Specifications

POWER	115 VAC or 230 VAC; 50/60 Hz
WEIGHT	12 pounds
OPERATING TEMPERATURE	10 ^{o}C to 40 ^{o}C
DIMENSIONS	17"(w) x 13"(d) x 7.5"(h)
SWEEP RATE	dual range: 0 to 99.99 mV/sec or 0 to 999.9 mV/sec
SWEEP RANGE	±9.999 Volts; set using push-button potentiometers
ELECTRODE CURRENT	1 Amp, maximum
OFFSET VOLTAGE	±9.999 Volts; set using push-button potentiometers
ELECTRODE VOLTAGE	±10 Volts
CURRENT CONVERTERS	7 ranges from 100 nA/V to 100 mA/V
INPUT IMPEDANCE	10^{12} ohms on reference potential follower
SMALL SIGNAL BANDWIDTH	greater than 10 kHz; –3dB typical
DUMMY RESISTORS	REF (100K), CE (2K), K1&K2 (1K)

Specifications are subject to change without notice.

Interface Cable Description

pin #	A-to-D board signal name	AFCBP1 signal name	direction of signal	description of signal (not all lines are used)
1	AI GND	AGND		analog ground (for board inputs)
2	AI GND	AGND		analog ground (for board inputs)
3	ACH00	SWEEP	CBP–>BOARD	output of CBP's internal sweep generator
4	ACH08	AGND		analog ground
5	ACH01	E1 OUT	CBP–>BOARD	potential of the K1 working electrode
6	ACH09	AGND		analog ground
7	ACH02	I1 OUT	CBP–>BOARD	current at the K1 working electrode
8	ACH10	AGND		analog ground
9	ACH03	E2 OUT	CBP–>BOARD	potential of the K2 working electrode
10	ACH11	AGND		analog ground
11	ACH04	I2 OUT	CBP–>BOARD	current at the K2 working electrode
12	ACH12	AGND		analog ground
13	ACH05	OL+	CBP–>BOARD	positive overload condition if this line higher than +7.5V
14	ACH13	AGND		analog ground
15	ACH06	OL–	CBP–>BOARD	negative overload condition if this line is less than –7.5V
16	ACH14	AGND		analog ground
17	ACH07	SW LIMIT	CBP–>BOARD	indicates the present target sweep limit (unused)
18	ACH15	AGND		analog ground
19	AI SENSE			
20	DAC0OUT	K1 INPUT	BOARD–>CBP	potential offset applied to K1 working electrode
21	DAC1OUT	K2 INPUT	BOARD–>CBP	potential offset for K2 *or* back panel rotator control
22	EXTREF			
23	AO GND	AGND		analog ground (for board outputs)
24	DIG GND	DGND		digital signal ground
25	ADIO0	ED0	BOARD<>CBP	used for encoded digital communication
26	BDIO0	ED4	BOARD<>CBP	used for encoded digital communication
27	ADIO1	ED1	BOARD<>CBP	used for encoded digital communication
28	BDIO1	ED5	BOARD<>CBP	used for encoded digital communication
29	ADIO2	ED2	BOARD<>CBP	used for encoded digital communication
30	BDIO2	ED6	BOARD<>CBP	used for encoded digital communication
31	ADIO3	ED3	BOARD<>CBP	used for encoded digital communication
32	BDIO3	ED7	BOARD<>CBP	used for encoded digital communication
33	DIG GND	DGND		digital signal ground
34	+5V			
35	+5V			
36	SCANCLK			
37	EXTSTRB			
38	STRT TRIG		CBP–>BOARD	signal to begin acquisition (sync with sweep start)
39	STOP TRIG		CBP–>BOARD	signal to halt acquisition (sync with sweep end)
40	EXTCONV			
41	SOURCE1			
42	GATE1			
43	OUT1	ED8	BOARD->CBP	strobes digital data sent from board to potentiostat
44	SOURCE2			
45	GATE2			
46	OUT2			
47	SOURCE5		CBP–>BOARD	strobes digital data sent from potentiostat to board
48	GATE5			
49	OUT5			
50	FOUT			

Note: All digital communication between the board and potentiostat is encoded using an error correction scheme to avoid miscommunication. A message packet sent to the potentiostat contains information regarding all front panel settings, potential limits and sweep rates for the analog sweep generator, and other important internal switch settings. The message returned by the potentiostat indicates the success or failure of the communications attempt and whether or not any message parameters are out of range. A software library written in the "C" language that handles communication with the bipotentiostat is available directly from Pine Instrument Company.

**Technical Reference Manual for
the ASCBDEV Developer's Kit**

**from
Pine Instrument Company
Grove City, Pennsylvania**

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Table of Contents

INTRODUCTION

SCOPE OF THIS MANUAL

This manual is meant for a person who is experienced in both electrochemistry and computer programming. It describes how to control the AFCBP1 Bipotentiostat using a library of low-level software functions written in the C programming language.

To make effective use of the information in this manual, you need to have a personal computer equipped with a suitable INTERFACE BOARD. The board should be connected to the 50-pin connector on the back side of the AFCBP1 Bipotentiostat. In addition, you should be familiar with the information in the manuals which accompany the INTERFACE BOARD, especially those which describe the NI-DAQ data acquisition library created by *National Instruments Corporation*.

This manual describes the use of the CBPCOM communications library developed for use with the AFCBP1 Bipotentiostat. As of this printing, the CBPCOM library supports the use of about ten different interface boards offered by *National Instrument Corporation*.

INTERNET RESOURCES

The latest versions of the *NIDAQ* device driver libraries can be downloaded from the *National Instruments Corporation* web site (www.natinst.com). The site also offers complete documentation and on-line help services for the *NIDAQ* library.

Pine Instrument Company also has a web site (www.pineinst.com) that offers up-to-date information about our products and software. Technical help may be requested via electronic mail (info@pineinst.com) as well.

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INTERFACE DESCRIPTION

The section of the manual gives a general overview of the connection between a personal computer system and the AFCBP1 BIPOTENTIOSTAT.

THE INTERFACE BOARD

The AFCBP1 Bipotentiostat was originally designed to work with the AT-MIO-16H-9 INTERFACE BOARD manufactured by *National Instruments Corporation* (Austin, TX). This board offers eight analog input (ADC) channels as required to monitor the eight analog signals generated by the AFCBP1 Bipotentiostat. In addition, it has two analog output (DAC) channels that can be used to apply potentials to each of the working electrodes. Finally, it has eight digital I/O lines that can be used for communicating with the 8032 CPU located within the AFCBP1 Bipotentiostat.

Several years ago, *National Instruments Corporation* discontinued the original AT-MIO-16H-9 board because it was based on the Am9513 timing chip (which had also become obsolete). Other boards based on this chip, including the AT-MIO-16L-9, the AT-MIO-16F-9, and the AT-MIO16-X were also discontinued.

A new series of boards, based on the custom DAQ-STC chip, was introduced by *National Instruments Corporation* to replace the older Am9513-based boards. This newer "E-series" offered superior timing capabilities and a wider selection of interface boards. *Pine Instrument Company* chose the AT-MIO-16E-10 as the replacement for the older AT-MIO-16H-9 board. Most software current offered by Pine supports either interface board.

More recent developments in the personal computer industry have required support for even more interface boards. The AT-MIO-16E-10 board is designed for a computer system with an ISA bus slot, and does not work with the newer style PCI slots. To provide support for the newer PCI style interface boards, the communications library described in the manual (CBPCOM) was designed to support the widest possible range of boards. The boards currently supported are listed below:

> ***ISA boards based on the older Am9513 chip:***
> AT-MIO-16H-9, AT-MIO-16L-9, AT-MIO-16F-5, AT-MIO-16X

> ***ISA boards based on the newer DAQ-STC chip:***
> AT-MIO-1E-10, AT-MIO-16E-2, AT-MIO-16E-1, AT-MIO-16XE-10

> ***PCI boards based on the newer DAQ-STC chip:***
> PCI-MIO-16E-1, PCI-MIO-16E-4, PCI-MIO-16XE-10

It is undoubtedly possible to use other types of boards with the AFCBP1 Bipotentiostat as well. If you wish to use some other board, make certain that it has enough ADC, DAC, and digital lines to fully communicate with the instrument. In addition, be prepared to make extensive modifications to the source code for the CBPCOM library to match the particular characteristics of your board. The information in this manual should help you in this process.

THE INTERFACE CABLE

The connection between the INTERFACE BOARD and the AFCBP1 Bipotentiostat is made via a 50-conductor INTERFACE CABLE. There are both analog and digital signal lines in this ribbon cable. The analog lines are used mainly to convey current and potential signals from the AFCBP1 Bipotentiostat to the INTERFACE BOARD. However, two of the analog lines are used to connect the DAC outputs from the INTERFACE BOARD to the K1 and K2 working electrode potential inputs. Finally, eight digital lines allow for control of the AFCBP1 front panel settings and the internal SWEEP GENERATOR.

A complete description of the 50-pin ribbon cable connector is given in Table I. Note that not all of the signal lines are used. Also note that if an E-series interface board is being used, then a 68-to-50 pin CABLE ADAPTER is required to connect the 50-pin ribbon cable to the 68-pin connector on the board. The CABLE ADAPTER maps the appropriate E-series signal lines to the correct positions on the 50-pin ribbon cable. The adapter is available from either *Pine Instrument Company* or *National Instruments Corporation*.

Table I: Cross-Index for Interface Cable Signal Lines

pin #	E-series board signal*	Am9513 board Signal	AFCBP1 signal	signal direction	signal description
1	AI GND	AI GND	AGND		analog ground (for board inputs)
2	AI GND	AI GND	AGND		analog ground (for board inputs)
3	ACH00	ACH00	SW	CBP–>MIO	output of CBP's internal sweep generator
4	ACH08	ACH08	AGND		analog ground
5	ACH01	ACH01	E1	CBP–>MIO	potential of the K1 working electrode
6	ACH09	ACH09	AGND		analog ground
7	ACH02	ACH02	I1	CBP–>MIO	current at the K1 working electrode
8	ACH10	ACH10	AGND		analog ground
9	ACH03	ACH03	E2	CBP–>MIO	potential of the K2 working electrode
10	ACH11	ACH11	AGND		analog ground
11	ACH04	ACH04	I2	CBP–>MIO	current at the K2 working electrode
12	ACH12	ACH12	AGND		analog ground
13	ACH05	ACH05	OH	CBP–>MIO	positive overload if greater than +7.5V
14	ACH13	ACH13	AGND		analog ground
15	ACH06	ACH06	OL	CBP–>MIO	negative overload if less than –7.5V
16	ACH14	ACH14	AGND		analog ground
17	ACH07	ACH07	SL	CBP–>MIO	present target sweep limit
18	ACH15	ACH15	AGND		analog ground
19	AI SENSE	AI SENSE			
20	DAC0OUT	DAC0OUT	K1 INPUT	MIO–>CBP	potential applied to K1 working electrode
21	DAC1OUT	DAC1OUT	K2 INPUT	MIO–>CBP	potential for K2 or back panel rotator control
22	EXTREF	EXTREF			
23	AO GND	AO GND	AGND		analog ground (for board outputs)
24	DIG GND	DIG GND	DGND		digital signal ground
25	ADIO0	ADIO0	ED0	MIO<>CBP	used for encoded digital communication
26	BDIO0	BDIO0	ED4	MIO<>CBP	used for encoded digital communication
27	ADIO1	ADIO1	ED1	MIO<>CBP	used for encoded digital communication
28	BDIO1	BDIO1	ED5	MIO<>CBP	used for encoded digital communication
29	ADIO2	ADIO2	ED2	MIO<>CBP	used for encoded digital communication
30	BDIO2	BDIO2	ED6	MIO<>CBP	used for encoded digital communicat
31	ADIO3	ADIO3	ED3	MIO<>CBP	used for encoded digital communication
32	BDIO3	BDIO3	ED7	MIO<>CBP	used for encoded digital communication
33	DIG GND	DIG GND	DGND		digital signal ground
34	+5V	+5V			
35	+5V	+5V			
36	SCANCLK	SCANCLK			
37	EXTSTROBE	EXTSTRB			
38	PFI0/TRIG1	STRT TRIG		CBP–>MIO	signals beginning of sweep program
39	PFI1/TRIG2	STOP TRIG		CBP–>MIO	signals the end of sweep program
40	CONVERT	EXTCONV			
41	GPCTR1 SOURCE	SOURCE1			
42	GPCTR1 GATE	GATE1			
43	GPCTR1_OUT	OUT1	XMT STROBE	MIO->CBP	strobes digital data sent to potentiostat
44	PFI5/UPDATE	SOURCE2			
45	PFI6/WFTRIG	GATE2			
46	STARTSCAN	OUT2			
47	GPCTR0 SOURCE	SOURCE5	RCV STROBE	CBP–>MIO	strobes digital data sent from potentiostat
48	GPCTR0 GATE	GATE5			
49	GPCTR0_OUT	OUT5			
50	FREQ_OUT	FOUT			

* Note: Pin numbers for E-series boards assume that a 68-to-50 pin CABLE ADAPTER is in use.

ANALOG INPUT CHANNELS

Pins 3 through 18 are used for analog input from the AFCBP1 Bipotentiostat to the INTERFACE BOARD. There are eight ADC channels on the board numbered zero through seven (7), and eight analog signals generated by the AFCBP1 are directly connected to these ADC channels. Table II shows the use for each of the eight ADC channels.

The NI-DAQ library provided by *National Instruments Corporation* offers a wide variety of data acquisition functions that can be used to monitor one or more of these ADC channels. The channel scan functions are especially useful for acquiring large amounts of data using a limited number of function calls.

Table II: Analog Signals Generated by the AFCBP1 Bipotentiostat

Channel Number	Signal Name (abbreviation)	Description
0	Sweep Output (SW)	This signal always reflects the output level of the AFCBP1's internal analog SWEEP GENERATOR.
1	K1 Potential (E1)	This signal output from the AFCBP1 represents the voltage at the K1 working electrode.
2	K1 Current (I1)	This signal is a voltage proportional to the current flowing at the K1 working electrode. To convert this voltage to a current value requires knowledge of the present setting of the CURRENT CONVERTER.
3	K2 Potential (E2)	This signal output from the AFCBP1 represents the voltage at the K2 working electrode.
4	K2 Current (I2)	This signal is a voltage proportional to the current flowing at the K2 working electrode. To convert this voltage to a current value requires knowledge of the present setting of the CURRENT CONVERTER.
5	Positive Overload (OH)	If the voltage signal on this channel is greater than +7.5V, then a positive overload condition exists.
6	Negative Overload (OL)	If the voltage signal on this channel is less than –7.5V, then a negative overload condition exists.
7	Sweep Direction (SL)	When an analog sweep is in progress, this signal reflects the limit potential towards which the sweep is presently heading. It can be used to distinguish between the various segments in the sweep program.

Of course, the most important analog input signals are E1, I1, E2, and I2 as these are directly related to what is going on in the electrochemical cell. The other signals are used to monitor the status of the AFCBP1 Bipotentiostat and its internal SWEEP GENERATOR.

Measuring Potentials

The 12-bit ADC circuitry on most interface boards allows analog input signals to be measured with a precision of one part in 4096. Each channel on the board can be set up to measure signal levels over a given voltage range. The most useful voltage ranges are ±10V, ±5V, and ±2.5V, although even more sensitive ranges are available. The voltage range for each channel is determined by the *gain setting* for that channel. The NI-DAQ software library offers functions that allow you to set the channel gains prior to acquiring data.

If you are monitoring an analog input signal using the ±10V range, then you can measure the signal with a precision of 4.88 mV. (This is computed by taking the full 20 volt input range and dividing it by 4096.) If you need to make measurements with more precision, then you must use a smaller input range.

Three of the supported boards offer 16-bit ADC circuitry. These boards are the AT-MIO-16X, the AT-MIO-16XE-10, and the PCI-MIO-16XE-10, all of which measure signals with a precision of one part in 65536.

Measuring Currents

When you are monitoring one of the current signals (I1 or I2), you must know both the ADC channel gain and the present setting of the CURRENT CONVERTER in order to translate the ADC signal level to a meaningful current value. Use the NI-DAQ library functions to set the channel gain, and use the CBPCOM library to set the CURRENT CONVERTER range.

Monitoring the Sweep Generator

If you use the AFCBP1's internal analog SWEEP GENERATOR during your experiment, you have the option of monitoring the sweep output (SW) directly using ADC channel zero. Of course, if you are applying the sweep output to one of the electrodes, simply monitoring either E1 or E2 usually provides the same information. However, the E1 and E2 signals may differ from the SW signal when an offset potential is being applied to either electrode.

The sweep output generally consists of repetitive sweeping back and forth between two sweep limits. The overall sweep program consists of one or more sweep segments with the direction of the sweep changing from one segment to the next. Depending on your particular application, it may be important for you to know exactly when the sweep direction changes. If this is the case, your program can monitor ADC channel seven (7), which provides a synchronization signal (SL) that is related to the sweep direction.

The SL signal line is held at the limit potential towards which the sweep is presently heading. Whereas the sweep output (SW) is essentially a triangle wave bounded by two limit potentials, the SL signal is a square wave bounded by the same two limit potentials. The SL signal and the SW signal are synchronized with each other so that when the sweep direction changes, the SL signal suddenly steps from one limit potential to the other.

Overload Conditions

The feedback circuitry at the heart of the AFCBP1 is prone to overloading or serious oscillations under certain experimental circumstances. The most common cause of an overload is when one of the CURRENT CONVERTERS is set at too sensitive a level for the amount of current flowing in the cell. The next most likely cause is when there is too great an impedance between the REF and CE inputs on the front panel. Oscillations can occur whenever rapidly changing waveforms are applied to a cell with peculiar impedance characteristics.

In any of the cases described above, the AFCBP1 warns you that it is in an overload state by activating the OVERLOAD indicator on the front panel. You can design your software to detect this condition by having it monitor ADC channels 5 and 6. If the signal on channel 5 exceeds +7.5 volts or if the signal on channel 6 falls below –7.5 volts, then an overload condition exists.

ANALOG OUTPUT CHANNELS

The INTERFACE BOARD provides two digital-to-analog channels (DACs) that can be used to output potentials to the AFCBP1. The two channels are channel zero and channel one. The output of channel zero is connected directly to the potential input for the K1 working electrode, and the output of channel one is connected directly to the K2 working electrode.

The potential actually applied to a working electrode is the sum of the appropriate DAC output potential, the SWEEP GENERATOR output (if active), and any externally applied potential signal present on the front panel "IN" connectors. Thus, you can use the DAC output channel as an offset potential in conjunction with the SWEEP GENERATOR output and/or some external waveform generator.

In most cases, you will want to disable the OFFSET VOLTAGE controls located on the front panel of the AFCBP1. The CBPCOM library allows you to turn off these controls in order to prevent the user from summing yet another offset potential to the working electrode.

The NI-DAQ library offers a variety of functions that you can use to apply potentials to the working electrodes. The simple **"AO_VWrite"** function can apply any potential between –10V and +10V to an electrode. More advanced waveform generation functions are offered by the NI-DAQ library if you wish to perform pulse or stairstep voltammetry experiments.

DIGITAL COMMUNICATIONS

The most complex aspect of controlling the AFCBP1 Bipotentiostat is establishing digital communications. Originally, the AFCBP1 was designed for use with the AT-MIO-16H-9 board. This board has only eight (8) digital I/O lines and a few triggering and strobe lines that can be used for handshaking. If you take a quick glance at the front panel of the AFCBP1, it is readily apparent that there are far more than just eight digital controls on it. Obviously, there are not enough digital lines to map each control to its own digital signal.

In order to control all aspects of the AFCBP1 along with its internal SWEEP GENERATOR, there had to be a way to transmit a large amount of digital information from the INTERFACE BOARD to the AFCBP1. It was decided that the best way to do this was to send the information as a message packet. Currently, this message packet consists of 62 octets (bytes) of information plus two additional checksum octets. (The checksum is used to check for transmission errors.)

The message packet is sent to the AFCBP1 one octet at a time using all eight digital lines as output lines. Each time an octet of information is placed on the output lines, a data strobe signal is sent to the AFCBP1 telling it to read the octet. The eight digital lines are on pins 25 to 32 of the INTERFACE CABLE. The strobe signal is sent on pin 43.

Upon receipt of the message packet, the AFCBP1 responds to any commands in the message and configures itself accordingly. It sends a brief reply message back to the INTERFACE BOARD. This reply is only eight (8) octets long. In order to receive this reply, the eight digital lines must be reconfigured for input (as soon as possible) after a message packet is sent to the AFCBP1. The reply message is read one octet at a time, and the AFCBP1 sends a strobe signal back to the INTERFACE BOARD (using pin 47) each time a octet is ready to be read.

Integer Representation within the AFCBP1 Bipotentiostat
The 8032 processor within the AFCBP1 Bipotentiostat represents 16-bit integers in "big-endian" form while *Windows*-based personal computers store such integers in "little-endian" form. As a result, all of the 16-bit integers in the message packet must be explicitly changed to big-endian form before transmitting the packet to the AFCBP1. Fortunately, this is a simple matter of swapping the order of the two octets that make up a 16-bit integer.

Generating Handshaking Strobes using NIDAQ
The strobes used for timing digital communications between the board and the AFCBP1 make use of the digital counters found on the Interface Board. There are remarkable differences between the older Am9513 counters and the newer DAQ-STC counters. The CBPCOM library deals with these differences by including two completely different sets of program functions, each designed to work with one of the two counters. The CBPCOM functions listed in the source code include either "Am9513" or "DAQSTC" in their names in order to explicitly indicate the timer chip for which they are intended.

On the older Am9513 boards, counter one (1) is used for sending strobes to the AFCBP1, and counter five (5) is used for receiving strobes. The NI-DAQ device driver functions used to control the counters on this chip are grouped together in the "CTR" family of functions. Consult the *NI-DAQ* documentation for more details on how to use these functions.

On the newer DAQ-STC based boards, general purpose counter one (GPCTR1) is used for sending strobes to the AFCBP1, and general purpose counter zero (GPCTR0) is used for receiving strobes. The NI-DAQ device driver functions used to control the counters on this chip are grouped together in the "GPCTR" family of functions. Consult the *NI-DAQ* documentation for more details on how to use these functions.

Communications Timing Parameters
Figure 1 and Table III summarize how the two strobe signals and the eight data lines are used during a typical packet transmission event. During periods of inactivity, the transmit strobe (XMT STROBE, pin 43) should be maintained in a logic HIGH (+5 V) state. A high-to-low transition on this line signals the AFCBP1 that a valid data octet is present on the digital lines. Likewise, the AFCBP1 normally maintains the receive strobe (RCV STROBE, pin 47) at a logic HIGH (+5V) state. A high-to-low transition on this line indicates that the AFCBP1 has placed a valid reply packet octet on the digital lines.

The very first octet of a message packet is sent to the AFCBP1 as follows. First, the digital lines on the INTERFACE BOARD are configured for output. Then, the data for the first octet is written to the digital output port on the board. Next, a high-to-low transition on the XMT STROBE line signals the AFCBP1 to read the octet. At this point, the AFCBP1 requires a certain amount of time (the XMT OCTET DWELL TIME, t_{X0}) to read and store the octet in its internal buffers.

Subsequent octets sent to the AFCBP1 are transmitted in the same fashion. The total time taken to transmit the entire 64 octet message packet must not exceed 500 milliseconds, or else the AFCBP1 flushes its input buffers and ignores the incomplete packet. This effectively places an upper limit on the interval between octets (XMT OCTET INTERVAL, t_{X1}) of about six milliseconds (6 msec).

The final octet in the message packet must remain on the digital output lines for a certain amount

of time (XMT OCTET DWELL TIME, t_{X0}) before switching the lines over for use as digital input lines. The delay between transmission of the message packet to the AFCBP1 and the reply packet from the AFCBP1 is about 18 milliseconds (RCV Reply Time, t_{R0}). Each of the eight octets in the reply packet is strobed by a high-to-low transition on the RCV STROBE line. Each reply octet remains on the digital lines for about 20 milliseconds (RCV OCTET DWELL, t_{R1}). The strobe signal is a LOW (zero volts) pulse lasting about 6.5 milliseconds (RCV STROBE DURATION, t_{R3}).

Receipt of the entire reply packet typically takes about 200 milliseconds. After that, the AFCBP1 still needs some additional time to process the message. For this reason, the overall interval between message packets should be at least 300 milliseconds (i.e., XMT PACKET INTERVAL, t_{X3}).

NOTE:	More recent versions of the CBPCOM library vary the length of the transmit strobe pulse to achieve the timing delays required for proper communication. The NIDAQ functions used to control and monitor the counters on the INTERFACE BOARD permit the duration of the HIGH and LOW parts of the XMT STROBE pulse to be of various lengths. By relying on these counters to keep time (rather than using arbitrary delay loops), the control of the communications timing is more deterministic and less prone to arbitrary latencies in code execution time.

Checksum Computation

There is always a chance that a message packet might be corrupted by electrical noise during transmission. The main purpose of the short reply message from the AFCBP1 is to verify that the message packet was transmitted without error. Each time the AFCBP1 processes an incoming message packet, it validates the packet using a checksum placed in the last two octets of the message packet. If the packet is invalid, then the reply from the AFCBP1 contains a code indicating an error condition.

The CRC checksum algorithm used by the CBPCOM library is somewhat complicated is implemented in the "PCOMM_XMT_COMPUTE_CRC_CHECKSUM" function. If you use this function without modification, you should have no trouble producing the proper checksum value to place at the end of a message packet.

If, however, you have to implement this function in another language or using a different programming platform, you will need to make certain that your function is producing the right checksum value. To help you in debugging your CRC checksum function, three complete message packets are given in Table IV in hexadecimal. These message packets, as shown, are ready for transmission to the AFCBP1 (that is, they contain big-endian integers and a properly computed checksum value.)

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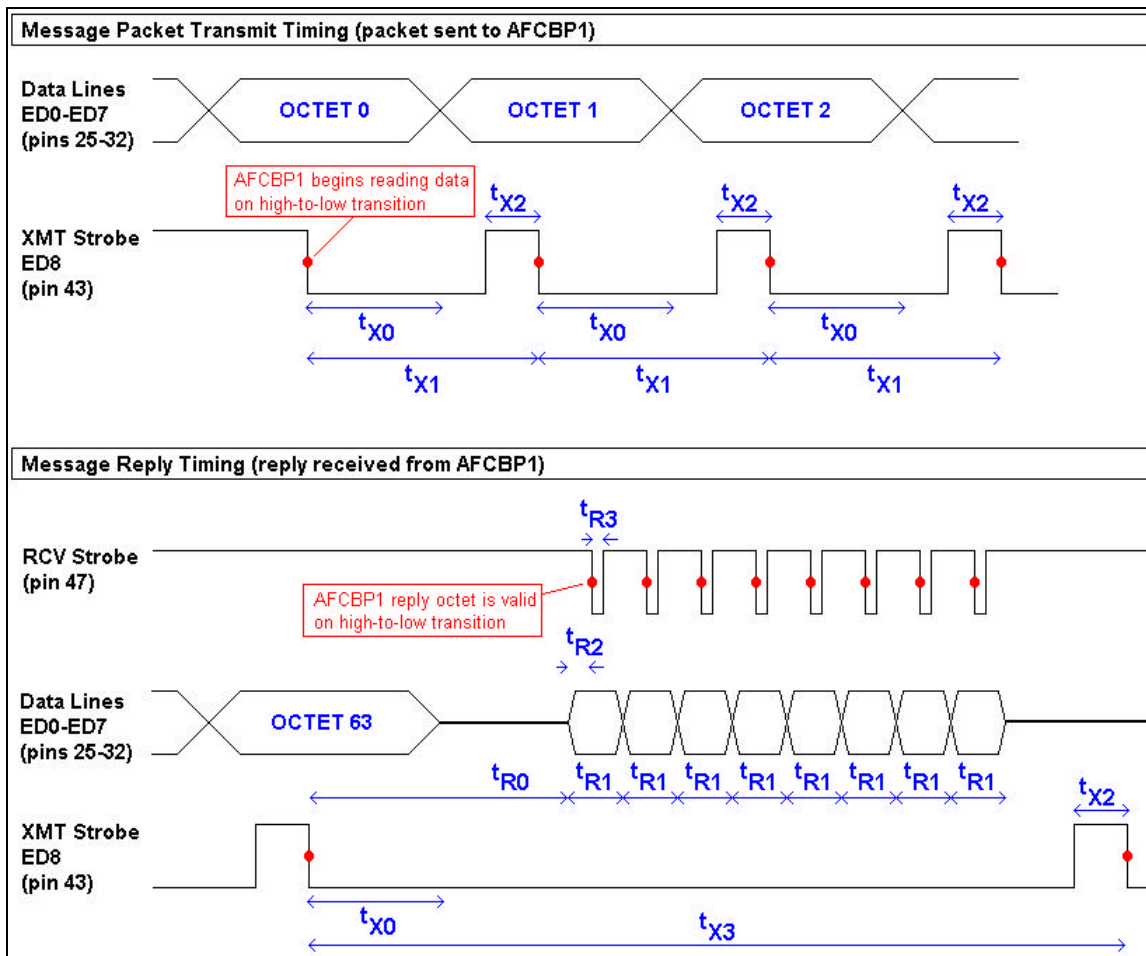
Figure 1: AFCBP1 Communications Timing Diagram

Table III: Critical Timing Parameters

Parameter	Symbol	Minimum	Typical	Maximum	Units
XMT OCTET DWELL	t_{X0}	150	150		μsec
XMT OCTET INTERVAL	t_{X1}	155	180	6000	μsec
XMT STROBE DURATION	t_{X2}	1	5		μsec
XMT PACKET INTERVAL	t_{X3}	300	300		μsec
RCV REPLY TIME	t_{R0}	18	18	20	msec
RCV OCTET DWELL	t_{R1}		20		msec
RCV OCTET LEAD TIME	t_{R2}		7		msec
RCV STROBE DURATION	t_{R3}		6.5		msec

Table IV: Sample Message Packets

Octet	Variable	Message #1	Message #2	Message #3
0	pcomm_Command	00	00	00
1		14	FF	14
2	pcomm_OpMode	00	00	00
3		00	00	00
4	pcomm_PosSweepRate	00	01	00
5		00	F4	00
6	pcomm_NegSweepRate	00	01	00
7		00	F4	00
8	pcomm_ManSweepDir	00	00	00
9		04	08	04
10	pcomm_NumLegs	00	00	00
11		00	07	00
12	pcomm_BeforeDelay	00	00	00
13		00	64	00
14	pcomm_AcqDelay	00	FF	00
15		01	FF	01
16	pcomm_K1OffsetState	00	00	00
17		02	02	02
18	pcomm_K2OffsetState	00	00	00
19		02	02	02
20	pcomm_K1Range	00	00	00
21		00	03	00
22	pcomm_K2Range	00	00	00
23		00	06	00
24	pcomm_K1SweepState	00	00	00
25		00	01	00
26	pcomm_K2SweepState	00	00	00
27		00	00	00
28	pcomm_GalPot	00	00	00
29		00	00	00
30	pcomm_OpenLoop	00	00	00
31		01	01	01
32	pcomm_DummyNormal	00	00	00
33		01	00	00
34	pcomm_SweepHold	00	00	00
35		01	00	01
36	pcomm_StopAtLower	00	00	00
37		01	01	01
38	pcomm_StopAtUpper	00	00	00
39		01	01	01
40	pcomm_AcqLength	00	FF	00
41		02	FF	02
42	pcomm_DisengageDelay	FF	FF	FF
43		FF	FF	FF
44	pcomm_SweepZero	00	00	00
45		01	01	01
46	pcomm_Range	00	00	00
47		00	00	00
48	pcomm_InitPot	00	00	00
49		00	00	00
50	pcomm_UpperLimit	00	00	00
51		01	00	01
52	pcomm_LowerLimit	FF	FD	FF
53		FF	44	FF
54	pcomm_FinalPot	00	FD	00
55		00	44	00
56	pcomm_NoConnect	00	00	00
57		01	00	00
58	pcomm_Unused1	00	00	00
59		00	00	00
60	pcomm_Unused2	00	00	00
61		00	00	00
62	pcomm_CheckSum	E0	1E	21
63		B4	CA	49

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COMMUNICATIONS LIBRARY

The CBPCOM communications library was developed by *Pine Instrument Company* to facilitate control of the AFCBP1 Bipotentiostat by programs written in the C programming language. The main function of this library is to provide a standard way to send message packets to the AFCBP1 over the digital I/O lines on the INTERFACE BOARD.

The library provides absolutely no data acquisition capability and no support for reading the analog input (ADC) channels. Ample support for data acquisition from the ADC channels is provided by the NI-DAQ library functions supplied by *National Instruments Corporation*. Similarly, no support is provided for analog output functions as the NIDAQ device driver library provides ample support for such functions.

The software interface to the CBPCOM library is a set of four public functions, a structure definition for the message packet, and a rather large set of constant definitions, all of which can be found in the "CBPCOMxx.H" header file. Many of the function and variable names begin with the "pcomm" prefix. This is a holdover from the earlier versions of the library which was itself named the "PCOMM" library.

THE FUNCTIONS

The public functions exported by CBPCOM 3.2 are described below. Further details as to how each function works may be gleaned by reading through the source code for the CBPCOM library (included on the diskette).

PCOMM_Initialize

This function should be called once to initialize the library. It causes the library to build an internal list of all of the NIDAQ devices presently installed on the computer system.

This function has no parameters and no return value.

PCOMM_SetDefaults

This function can be used to set all of the PCOMM variables in a message packet to their default values. This provides a good starting point from which to build your own message packet. Use of this function is optional, and it is provided for convenience only.

The only parameter passed to this function is a pointer to the message packet's memory buffer. It is assumed that the memory for the buffer has already been allocated.

This function has no return value.

PCOMM_ResetBoard

This function resets the interface board and sends a "buffer flushing" pulse to the AFCBP1. This function requires one parameter.

The only parameter is a 16-bit integer which specifies the NI-DAQ device number for the INTERFACE BOARD. This device number is assigned to the board during the board installation process (consult the board manual for details).

This function returns an error code indicating the success or failure of the operation. All possible error codes are defined in the CBPCOMxx.H header file. Error codes with values less than –59 are NIDAQ device driver library error codes.

PCOMM_Hello
This function should be called once to set up initial communication with the AFCBP1. This function initializes the interface board (by calling ***PCOMM_ResetBoard***), sends a message packet to the AFCBP1, and waits for a reply. This function requires two parameters.

The first parameter is a 16-bit integer which specifies the NI-DAQ device number for the INTERFACE BOARD. This device number is assigned to the board during the board installation process (consult the board manual for details).

The second parameter is used to return the version number for the EPROM within the AFCBP1. This parameter is a 16-bit integer. The current EPROM version number is 2.02, so this parameter should be equal to 0202 (hexadecimal) upon return.

This function returns a boolean result which indicates whether or not the attempt to communicate with the AFCBP1 was successful.

PCOMM_SendMessage
This function sends a message packet to the AFCBP1. This is the most important and most complicated function in the CBPCOM library. This function requires four parameters.

The first parameter is a 16-bit integer which specifies the NI-DAQ device number for the INTERFACE BOARD. This device number is assigned to the board during the board installation process (consult the board manual for details).

The second parameter is a pointer to the message packet that is to be sent to the AFCBP1.

The third parameter is an integer representing the number of attempts that should be made in trying to send the packet to the AFCBP1. A good value for this parameter is three (3).

The fourth parameter is used to return the version number for the EPROM within the AFCBP1. This parameter is a 16-bit integer. The current EPROM version number is 2.02, so this parameter should be equal to 0202 (hexadecimal) upon return.

This function returns an error code indicating the success or failure of the transmission operation. All possible error codes are defined in the CBPCOMxx.H header file. Error codes with values less than –59 are NIDAQ device driver library error codes.

PCOMM_SetCommuncationsTiming
This function allows the caller to make changes to the fundamental communications timing parameters which govern message packet transmission. Normally, this function does not need to be used. This function requires three parameters.

The first parameter is a 16-bit integer which specifies the NI-DAQ device number for the INTERFACE BOARD. This device number is assigned to the board during the board installation process (consult the board manual for details).

The second parameter is a constant defining the communications timing parameter to be changed. These constants are defined in the CBPCOMxx.H header file.

The third parameter is the new value for the commuications parameter.

This function has no return value.

PCOMM_GetCommuncationsTiming
This function allows the caller to obtain the value of one of the fundamental communications timing parameters which govern message packet transmission. Normally, this function does not need to be used. This function requires two parameters.

The first parameter is a 16-bit integer which specifies the NI-DAQ device number for the INTERFACE BOARD. This device number is assigned to the board during the board installation process (consult the board manual for details).

The second parameter is a constant defining the communications timing parameter to be changed. These constants are defined in the CBPCOMxx.H header file.

This function returns an unsigned 32-bit integer that is the present value of the timing parameter.

PCOMM_ResetCommuncationsTiming
This function allows the caller to reset all of the fundamental communications timing parameters which govern message packet transmission. Normally, this function does not need to be used. This function requires one parameter.

The only parameter is a 16-bit integer which specifies the NI-DAQ device number for the INTERFACE BOARD. This device number is assigned to the board during the board installation process (consult the board manual for details).

This function has no return value.

GENERAL VARIABLES

The message packet can be viewed as a series of 64 octets, a set of 32 integers (16-bit), or as a set of 32 "pcomm" variables. The CBPCOM_MSG_PKT data structure is a union which permits the message packet to be treated in any of these three ways. In the description that follows, the message packet is described in terms of its 32 constituent "pcomm" variables. These 32 variables provide complete control of the AFCBP1 Bipotentiostat.

The many constants which define the allowed values for the various pcomm variables are listed in the CBPCOM31.H header file.

pcomm_Command
This variable defines the action that you expect the AFCBP1 to take when it receives the message packet. Three commands are recognized: CBP_START_EXPERIMENT (10), CBP_STOP_EXPERIMENT (20), and CBP_IDLE_COMMAND (255). If the command code is invalid, then error code CBP_COMMAND_ERROR (-1) is returned.

If *pcomm_Command* is CBP_START_EXPERIMENT (10), then the AFCBP1 BIPOTENTIOSTAT will initiate the sweep once the message is processed. If the *pcomm_Command* is CBP_STOP_EXPERIMENT (20), then the AFCBP1 BIPOTENTIOSTAT will halt any sweep program that may already be in progress.

The CBP_IDLE_COMMAND (255) can be used when you want to send a message to the AFCBP1 without starting or stopping the sweep program. This does not necessarily mean that the sweep will be unaffected. If the message changes any of the sweep parameters, then the SWEEP GENERATOR will respond accordingly, even if *pcomm_Command* is equal to CBP_IDLE_COMMAND.

The very first message sent to initialize the AFCBP1 should have the *pcomm_Command* variable set equal to CBP_STOP_EXPERIMENT (20). This halts any sweep program that the SWEEP GENERATOR might presently be executing. As part of this same initial message to the AFCBP1, you should make certain that the variable *pcomm_SweepHold* is set to HOLD (1) so that the sweep doesn't just start over again. You may also wish to set the variable *pcomm_SweepZero* to TRUE (1) to assure that the sweep output is zero volts. If you do not intend to use the sweep at all, then every message you send to the AFCBP1 should always have *pcomm_SweepHold* and *pcomm_SweepZero* set in this manner.

To define and then execute a sweep program, you must send *two* messages to the AFCBP1. The first message should define the sweep program using the various global "pcomm" variables, and the *pcomm_Command* variable should be set to the CBP_IDLE_COMMAND (255). This first message gives the AFCBP1 time to adjust the internal SWEEP GENERATOR settings appropriately. Then, send a second *identical* message, but change the value of the *pcomm_Command* variable to CBP_START_EXPERIMENT (10). The sweep program will commence once the AFCBP1 has processed the second message.

pcomm_GalPot
This variable determines whether the K1 electrode circuit operates as a potentiostat or a galvanostat. The two valid settings for this variable are zero (0) for potentiostat mode or one (1) for galvanostat mode. Any other value results causes a CBP_GALPOT_ERROR (-15).

pcomm_Open
This variable determines whether the AFCBP1 operates in "open-loop" potential mode (see the AFCBP1 manual for more details). The two valid settings for this variable are CBP_OPENLOOP_CLOSED (1) for normal operation or CBP_OPENLOOP_OPEN (0) for "open-loop" mode. Any other value causes a CBP_OPEN_ERROR (-16).

pcomm_DummyNormal
This variable determines whether the AFCBP1 is connected to the external cell (normal) or an internal resistor network (dummy). The two valid settings for this variable are as follows:

CBP_DUMMYNORMAL_NORMAL (0) for normal operation, or
CBP_DUMMYNORMAL_DUMMY (1) for dummy mode.

Any other causes a CBP_DUMMYNORMAL_ERROR (-17).

pcomm_NoConnect
This variable determines whether the AFCBP1 is connected to the internal dummy cell or the

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actual external electrochemical cell *during the sweep program*. If the value of this variable is set equal to CBP_NOCONNECT_DUMMY_CELL (1), then the AFCBP1 uses the dummy cell. If it is set equal to CBP_NOCONNECT_EXTERNAL_CELL (0), then it uses the external cell. Any other value causes a CBP_NOCONNECT_ERROR (-28).

pcomm_K1SweepState, pcomm_K2SweepState

These variables determine whether or not the sweep output is applied to the K1 and/or K2 working electrode circuits. Setting either of these variables equal to one (1) causes the sweep to be applied to the appropriate electrode (K1 or K2). Setting both of them equal to one causes the sweep to be applied to both working electrodes.

If you will not be using the sweep, you should set both of these variables equal to zero. Note that when the sweep output is applied to an electrode circuit, it is summed with any offset potentials that are being applied at the same time.

If either variable is set to a value other than zero or one, a CBP_SWEEPSTATE_ERROR (-13) code is returned.

pcomm_K1OffsetState, pcomm_K2OffsetState

These variables determine whether or not the Offset Voltage controls physically located on the front panel of the AFCBP1 are active. Normally, you should deactivate these controls by setting these variables to CBP_OFFSETSTATE_DISABLED (2).

If for some reason, you want to make use of these controls, you can set this variable to either CBP_OFFSETSTATE_POSITIVE (1) or CBP_OFFSETSTATE_NEGATIVE (0) to apply either a positive or negative offset to the electrode potential.

Any other value for these variables causes a CBP_OFFSETSTATE_ERROR (-10).

pcomm_K1Range, pcomm_K2Range

These variables are used to set the CURRENT CONVERTER ranges for the K1 and K2 working electrode circuits on the AFCBP1 front panel. There are seven different ranges numbered zero through six (6) as summarized in Table IV.

Table IV: Current Converter Ranges

range number	CURRENT CONVERTER
0	100 mA / V
1	10 mA / V
2	1 mA / V
3	100 µA / V
4	10 µA / V
5	1 µA / V
6	100 nA / V

SWEEP GENERATOR VARIABLES

pcomm_PosSweepRate

This variable is used to specify the sweep rate whenever the sweep is heading toward more positive (anodic) potentials. The value of this 16-bit integer must be non-zero, it must be a multiple of five (5), and it must be no higher than 9995. The sweep rate is specified in millivolts per second. If this variable is less than zero or greater than 10000, then a CBP_SWEEPRATE_ERROR (-3) is returned. The communications library does not check to make sure the value is a multiple of five.

pcomm_NegSweepRate

This variable is used to specify the sweep rate whenever the sweep is heading toward more negative (cathodic) potentials. The value of this variable is subject to the same constraints as the *pcomm_PosSweepRate* variable described above.

pcomm_ManSweepDir

This variable is used to control the initial direction of the sweep. The two useful settings for this variable are CBP_SWEEPDIR_DOWN (8) for sweeping *down* towards more negative (cathodic) potentials or CBP_SWEEPDIR_UP (4) for sweeping *up* towards more positive (anodic) potentials. If the value of this variable is less than zero or greater than nine (9), it causes a CBP_MANSWEEPDIR_ERROR (-5).

pcomm_SweepHold

This variable determines whether the sweep output is held constant or allowed to continue running. The two valid settings for this variable are zero (0) which allows the sweep to continue or one (1) which *holds* the sweep output constant. Any other value causes a CBP_SWEEPHOLD_ERROR (-18).

If you will not be using the sweep in your experiment, you should set this variable to one (1). If you wish to start the sweep, then you should set the variable to zero (0) and also set the *pcomm_Command* to START_EXPERIMENT.

pcomm_StopAtLower, pcomm_StopAtUpper

These variables determine whether or not the sweep stops when it reaches a limit potential. The two valid settings for this variable are zero (0) which allows the sweep to continue or one (1) which stops the sweep at the limit. Any other value causes a CBP_LIMITSTOP_ERROR (-19).

These variables are not very useful and should be set equal to one (1) for normal operation.

pcomm_SweepZero

This variable is used to zero the sweep output. The two valid settings for this variable are zero (0) which allows the sweep to operate normally or one (1) which forces the sweep output to zero volts. Other values cause a CBP_SWEEPZERO_ERROR (-23).

If you will not be using the sweep in your experiment, you should set this variable to one (1). If you will be using the sweep, then you can still set this variable to one (1) if you want to assure that the sweep starts from zero volts.

pcomm_InitPot

This variable is used to set the initial potential for the sweep program. It is generally best to start the sweep program from zero volts by using a value of zero (0) for this variable. If you need the

sweep to start from some value other than zero, you can use an offset potential in conjunction with the sweep output to achieve the desired result.

pcomm_UpperLimit, pcomm_LowerLimit

These variables are used to specify the upper and lower sweep limit potentials. The value of these 16-bit integers must be in the range from -9995 to 9995. The potentials are specified in millivolts. If one of the potentials is out of range, the a CBP_SWEEPLIMIT_ERROR (-26) is returned.

The value of *pcomm_UpperLimit* must be at least 10 millivolts greater than the value of *pcomm_LowerLimit* for proper sweep performance. If the value of *pcomm_LowerLimit* is greater than *pcomm_UpperLimit*, it causes a CBP_SWEEPLIMIT_INVERSION_ERROR (-30) to be returned.

pcomm_FinalPot

This variable must be set equal to the potential at which the sweep is to stop. Normally, you will set the final potential equal to one of the two limit potentials (upper or lower, depending on the sweep direction). However, if you wish, you can have the sweep stop at some potential other than the usual limit potential.

The value of this 16-bit integer must be in the range from -9995 to 9995. The potential is specified in millivolts. If the potential is out of range, it causes a CBP_FINALPOT_ERROR (-27) to be returned.

In order to assure proper sweep operation, you must make sure that the final potential is valid. For example, if you know that the very last sweep segment in the sweep program starts at 800 mV and heads in a negative direction, then you should set the final potential to a value less than 800 mV.

pcomm_NumLegs

This variable specifies the total number of sweep segments in the sweep program. It should be set to zero if you will not be using the sweep. Otherwise, any value between 1 and 60,000 is acceptable. Values outside this range cause a CBP_NUMLEGS_ERROR (-6).

TIMING VARIABLES

pcomm_BeforeDelay

This variable specifies a delay period before the sweep program begins. If this variable is set equal to zero, then the AFCBP1 starts the sweep as soon as possible after receiving the START_EXPERIMENT command. Otherwise, it waits for a brief period of time before starting. The value of this variable determines the length of the delay period. The quantity of time is given in increments of 10 milliseconds. For example, if you set this variable equal to 200, then the delay period is two seconds long.

This variable must be set to a value in the range from zero to 60,000. Thus, the longest delay period is ten minutes. Values outside this range a CBP_BEFOREDELAY_ERROR (-7).

pcomm_AcqDelay

At some point during the sweep program, the AFCBP1 sends a triggering pulse to the INTERFACE BOARD. This pulse is sent on pin 38 and is meant to be used as a "start trigger" for the data acquisition process. The *NI-DAQ Function Reference Manual* describes how to set up the data

acquisition functions so that they wait for such an external trigger. Thus, you can use this start trigger to synchronize data acquisition with the sweep program.

If the *pcomm_AcqDelay* variable is set equal to 65535 (which is 0xFFFF hexadecimal), then the trigger pulse is sent at exactly the same time that the sweep program starts.

If the *pcomm_AcqDelay* variable is set to some value less than 65535, then the trigger pulse is sent at a given amount of time after receipt of the START_EXPERIMENT command. This quantity of time is given in increments of 10 milliseconds. For example, if you set this variable equal to 200, then the time period is two seconds long.

If the value of *pcomm_AcqDelay* is out of range, it causes a CBP_ACQDELAY_ERROR (-8).

pcomm_AcqLength

At some point after the start trigger, the AFCBP1 sends a second triggering pulse to the INTERFACE BOARD. This pulse is sent on pin 39 and can be used as a stop trigger for halting data acquisition, if desired. The *NI-DAQ Function Reference Manual* describes how to set up the data acquisition functions so that they look for such an external stop trigger.

If the *pcomm_AcqLength* variable is set equal to 65535 (which is 0xFFFF hexadecimal), then the trigger pulse is sent at exactly the same time as the sweep program stops.

If the *pcomm_AcqLength* variable is set to some value less than 65535, then the *stop trigger* is sent at a given amount of time after *start trigger*. This quantity of time is given in increments of 10 milliseconds. For example, if you set this variable equal to 200, then the time period is two seconds long.

An out of range value causes a CBP_ACQLENGTH_ERROR (-21).

pcomm_DisengageDelay

This variable determines whether or not the AFCBP1 automatically switches to the internal dummy cell after the stop trigger is sent. If you set the variable equal to 65535 (which is 0xFFFF hexadecimal), then the cell is not automatically switched to the dummy cell.

If this variable is set to some value less than 65535, then AFCBP1 switches to the internal dummy cell a certain amount of time after the *stop trigger*. This quantity of time is given in increments of 10 milliseconds. For example, if you set this variable equal to 200, then the time period is two seconds long.

An out of range value causes a CBP_ DISENGAGEDELAY_ERROR (-22).

RETURN CODES

If an error occurs during a call to PCOMM_Hello or PCOMM_SendMessage, then an error code is returned to the caller. The most commonly encountered error codes are described below:

CBP_DEVICE_NUMBER_ERROR (-29)
This code indicates that the NIDAQ device number passed to the function was invalid.

CBP_DEVICE_UNSUPPORTED (-35)
This code indicates that the requested NIDAQ device is an interface board that is not supported by this version of the CBPCOM library.

CBP_XMT_CHECKSUM_ERROR (-40)
This code indicates that the AFCBP1 received a corrupted message packet.

CBP_RCV_PKT_SHORT (-41)
This code indicates that the reply from the AFCBP1 did not contain enough octets.

CBP_RCV_PKT_LONG (-42)
This code indicates that the reply from the AFCBP1 contained too many octets.

CBP_RCV_PKT_TIMEOUT (-43)
This code indicates that no reply packet was received from the AFCBP1. The most common causes of this error are a disconnected interface cable or when the AFCBP1 is not in EXTERNAL control mode.

SOURCE CODE DISKETTE

The source code for several versions of the CBPCOM library are included on the source code diskette. It is recommended that you use the most recent version; the older versions are provided for backward compatibility only. This library is written using the C programming language. It is the very same library used by *Pine Instrument Company* to develop software for the AFCBP1.

The library consists of five different files. Four of these files are header files (CBPCOMxx.H, CRCTABLE.H, NIBOARDS.H, and SOMETYPE.H), and the fifth file (CBPCOMxx.C) contains the actual lines of code. These files are shipped to you on diskette along with this manual.

The CBPCOMxx.H header file contains important definitions and structure declarations that describe the message packet. Error code values are also defined in this file.

The CRCTABLE.H header file contains a large array of integer values used to generate CRC checksums.

The NIBOARDS.H header file contains the NIDAQ code numbers for all of the interface boards manufactured by *National Instruments Corporation*.

The SOMETYPE.H header file contains abbreviations for common C data types.

Using Other Languages
Pine Instrument Company does not directly support the use of languages other than the C language. However, the information in these source code listings should be sufficient for the average programmer to develop their own library using another language.

Equipment Needed:
- Bi-potentiostat test kit. Includes 2 sets of "dummy" electrode resistors (10 ohm and 1 ohm)
- Stopwatch
- 4 ½ digit digital voltmeter

Visual Inspection:
- Front panel solder joints ok.
- Front panel switches work properly (not sticking).
- Tighten screws on power transistors/ heat sink on rear panel.
- Tighten screws on terminal block.
- Tighten all screws on the enclosure, holding boards, and other case mounted components.

Test Procedure:
1. Program EPROM

2. Measure the resistance on all 4 power supplies on micro board. All should be > 1k ohms.
   +5VDC across C30 on micro board
   +/- 25VDC, +/- 15VDC, +/- 10VDC from analog common to J7 on the micro board.

3. Attach the power cord and measure resistance across power cord with the power switch on.
   Input module at 120VAC   5 ohms   (older transformers on repairs   10 ohms)
   "   " at 240VAC   20 ohms (older transformers on repairs   36 ohms)

4. Disconnect all 3 connectors going from the other 2 boards to the micro board. Apply 115VAC slowly while monitoring 5VDC power supply. Check other 3 power supplies. Turn power off. Repeat while applying 240VAC (change input module accordingly). Return to 120VAC setting.

5. Verify the pin to pin wiring is correct on the cable going from J7 on the power supply board to J11 on the analog board, then reconnect the 3 connectors to other boards and re-check all 4 power supplies.

6. With the meter at R1 of the power supply board, adjust P1 for 10.010VDC
   With the meter at R2 of the power supply board, adjust P2 for 10.010VDC

7. Check all front panel pots for accuracy. (Gnd to wiper)
   2 limit pots and 2 offset pots, +/- 50mv max
   Sweep pot +/- 5mv max (adjust P3 for 1 volt on pin 3 of the pot)

8. Check all front panel switches and LEDs for proper operation.

9. Measure voltage from earth gnd to DC gnd. Must be less than 0.1VDC.

10. With ZERO switch "on", SWEEP switch on "hold", adjust P5 for 0.000VDC at the SWEEP OUTPUT jack.

11. With ZERO "on", SWEEP RATE at 9999 mv/sec and RANGE switch at 1000mv/sec, adjust P3 to obtain +/- 0.9766VDC at U19, pin 6. Change sweep direction back and forth and balance readings at U19, pin 6.

12. Set UPPER LIMIT pot to +2.5V and LOWER LIMIT pot to –2.5V. Set ZERO to "off" and SWEEP to "run". Adjust P10 for a 5 sec. sweep (use stopwatch). CW will shorten time.

13. Set SWEEP RATE to 9999mv/sec, RANGE to 100mv/sec, ZERO to "on". Adjust P4 to obtain +/- 0.09766VDC at U19 pin 6. Change sweep direction back and forth and balance readings.

14. Set UPPER LIMIT pot to +100mv and LOWER LIMIT pot to –100mv, RANGE to 1000mv/sec, SWEEP RATE to 500mv/sec, ZERO to "off", SWEEP to "run". Using the STOP AT LIMIT switches, sweep to one limit and note the voltage at the SWEEP output jack, then sweep to the other limit and note that voltage. Adjust P6 for equal voltages. (CCW lowers upper limit)

K1 ELECTRODE   (Make sure K2 is off and sweep voltage is off)

15. Set K1 OFFSET VOLTAGE pot to 1.000V. CURRENT CONVERTER to 100ua/V. With meter at I1, adjust P8 for equal voltages when switching from + and – offset.

16. With K1 OFFSET VOLTAGE pot to 0V, +/- switch "off", adjust P7 to obtain 0.000V at I1.

17. Check all current converter settings for + and – balance at I1. Set offset at 1mv at the lowest setting then increase to 10mv, 100mv, etc.. as you go up.

K2 ELECTRODE   (Make sure K1 is off and sweep voltage is off)

18. Set K2 OFFSET VOLTAGE pot to 0V and +/- switch to "off". Set CURRENT CONVERTER to 10ua/V. With meter at I2, adjust P9 for 0.000VDC.

19. Repeat step 17 for K2.

OPERATION CHECK LIST

20. High current check. Install 10 ohm resistors and switch to "Normal" on electrode switch. Set OFFSET VOLTAGES to 1.000V and CURRENT CONVERTERS to 10ma/V. Measure voltages at E1, I1, E2, I2.   E1, E2 = 1VDC   I1, I2 = 10VDC. Check + and – for balance.

21. Install 1 ohm resistors. Set CURRENT CONVERTERS to 100ma/V. E1, E2 = 1VDC   I1, I2 = 10VDC. Check + and - balance.

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22. With 1 ohm resistors installed, measure voltages across the resistors in the K1 and K2 jacks. Should be about 1VDC. (check +/- balance)

23. OVERLOAD check. Return to "dummy" electrodes. Set K1 CURRENT CONVERTER to 10ua/V and OFFSET VOLTAGE pot to 100mv. Monitor I1 voltage and OVERLOAD LED while you increment OFFSET VOLTAGE by 1mv steps. LED should light when I1 voltage is between 10.2VDC and 11.2VDC. Check + and – offset. Repeat for K2.

24. Check sweep to K1 and K2. Set SWEEP RATE to 9999 and RANGE to1000mv/sec. Set UPPER LIMIT pot to 500mv. Set UPPER STOP LIMIT switch to "on", SWEEP to "run", and ZERO to "off" (sweep output should be 0.5VDC). With meter at E1, turn K1 SWEEP VOLTAGE switch "on". E1 should read 0.5VDC. Jumper SWEEP OUTPUT jack to K1 IN jack. E1 should read 1.0VDC. Repeat for K2.

25. GALVANOSTAT mode check. Change mode switch to GALVANOSTAT. Set K1 offset voltage to 100mv. Voltage at I1 should be 0.1VDC at current converter settings of 10ma, 1ma, and 100ua. Voltage at E1 should increase by 10X (.01, 0.1, 1.0) as you go up the 3 current converter settings. Check at + and – offset voltage.

26. Check OPEN switch. With voltages at E1, I1, and E2 jacks, push OPEN switch. Voltages should go to 0VDC.

27. Calibrate panel DVM to match bench meter. Choose a medium voltage (3 to 5V) to calibrate. Adjustment pot on back of panel DVM. Check + and -.

28. Null DAC outputs.
    Adjust P11 for 0.000VDC at U37, pin 1.
        P12 for 0.000VDC at U38, pin 1.

29. Setup unit as follows and burn-in overnight on bench.

    ELECTRODE to "Dummy"   MODE to "pot"   SWEEP RATE to 9999
    RANGE to 1000mv/sec   ZERO at "off"   SWEEP to "run"
    UPPER SWEEP LIMIT pot to +3VDC   LOWER SWEEP LIMIT pot to –3VDC
    K1 OFFSET pot to +100mv   K2 OFFSET pot to –100mv
    K1, K2 CURRENT CONVERTERS to 1ma/V   K1, K2 SWEEP VOLTAGE to "on"

30. Repeat steps 6-28 after burn-in.

31. Test communication to computer and calibrate DAC sweep rate.

FINAL INSPECTION

- Make sure all connectors are in place and latched.
- Check wires to back compensation jacks. Pins 1 and 2 (grn and blk) to Rotator Output. Pins 3 and 4 (blue) to K2. Pins 6 and 7 (yel) to K1. Pins 9 and 10 (org) to GAL.
- Serialize and fill out Potentiostat checklist.

Rev. 0 was 1/24/96 C:\filer\procedur\cbp-1.cal
12/16/02   JS   Added Step 1. – Program EPROM, chg'd "pot" to "switch", Step 8

−**Table of Contents**

Troubleshooting CBP to PC Communication

A walkthrough guide to solving CBP bipotentiostat communication issues. You may download a PDF version of this troubleshooting guide.

This technical notes addresses communication issues that can arise when using the Pine Research Instrumentation AFCBP1 bipotentiostat interfaced to a PC, which controls the bipotentiostat through AfterMathTM Data Organization Software. There are several layers of software and hardware between AfterMath and the CBP bipotentiostat. Each layer must be functioning properly for AfterMath to communicate correctly and efficiently with the potentiostat. When troubleshooting communication issues, a systematic approach must be used to diagnose the problem. Refer to this technical note for the suggested approach.

DEVICE DRIVER SOFTWARE

The proper device driver software must be installed on the computer. The device driver is from National Instruments and is called "DAQmx". Older versions of the driver were called "NIDAQ". These drivers are available as (very large) downloads from National Instruments web site (http://www.ni.com/white-paper/6909/en); however, most customers should have the device driver on a DVD that was shipped to them along with the interface board.

1. Pine maintains links to the National Instruments download area (and advice about which versions of the device driver to use) on the following web page: http://www.voltammetry.net/pine/afcbp1/driver
2. An easy way to tell if DAQmx is present on a computer system is to look for a utility program called "MAX" (which stands for Measurement and Automation Explorer). This is usually on the desktop of any computer which has DAQmx installed. If not, it should be located in the START menu under the "National Instruments" folder.

PROPER BOARD INSTALLATION

The physical interface board must be installed in the computer in a full-size PCI slot. Sometimes, after a computer has been moved, the interface board may partially slide out of the slot. Always check to assure that the board is secured in the PCI slot and held in place by a screw.

1. When installing a new board in a new computer, it is generally best to first install the DAQmx driver, then turn off the computer, then install the interface board, and then turn the computer back on. Doing the steps in this sequence helps to assure that Windows properly recognizes the new board after it is installed.

WINDOWS RECOGNITION OF INTERFACE BOARD

Confirm that Windows recognizes and can communicate with the interface board. This is done by using the Device Manager (CONTROL PANEL □ SYSTEM □ DEVICE MANAGER) to look at a list of the hardware devices presently installed on the computer. The interface board should appear somewhere in the list. If it doesn't, then Windows can't see the board, which means that DAQmx can't see the board, which means that AfterMath can't see the board.

1. An example of how the interface board appears in the Device Manager can be seen here: http://www.voltammetry.net/windows/device_manager
2. If Windows stubbornly refuses to see the board, it is sometimes necessary to remove the board, restart Windows, shut down Windows, reinstall the board in a (preferably different) PCI slot, and restart Windows.
3. Windows can sometimes be tricked into "rediscovering" the board by using the "Scan for Hardware Changes"

option in the "Actions" menu of the Device Manager.

DAQmx COMMUNICATION TO INTERFACE BOARD

Confirm that "DAQmx" can "see" the interface board. This is done by using the MAX utility (Measurement and Automation Explorer). The interface board should appear as one of the attached devices (often under the "Data Neighborhood" or "Legacy Devices" category).

1. There are many different versions of the MAX utility. The link below gives an overview of what actions can be accomplished using the MAX utility: http://www.ni.com/white-paper/4638/en
2. It is possible to use the "Test Panels" feature of the MAX utility to see if the board is "awake" or malfunctioning. Typically, running one of the "Counter" tests is the easiest approach. Just turn on one of the counters (Counter 0 or Counter 1) and a big green indicator on the Test Panel should turn on. This is a pretty good sign that the board is alive and well.

AfterMath COMMUNICATION WITH AFCBP1

Confirm that AfterMath can "see" the interface board. When AfterMath starts, it senses the presence of the installed board and a "Pine AFCBP1" entry will appear in the lower-left pane of the AfterMath window. AfterMath will attempt to cold start the instrument and establish some initial "idle" conditions.

1. If AfterMath can see the AFCBP1, but it is not possible to perform any experiments, then a "permissions file" is probably missing from the AfterMath installation. Permissions files can be obtained by contacting Pine. Permissions files are usually located in the folder shown at the link: http://www.voltammetry.net/pine/aftermath/user/activation.
2. If AfterMath appears to be in a continuous loop, repeatedly trying to "cold start" the instrument, then the cable is probably loose, or the instrument is not in the proper "External Control" state (see below).

CABLE CONNECTIONS

Verify that the interface cable is secure on both ends. It is very common after moving the instrument or the computer for the cable to come loose from one of the two end connectors.

1. If the cable is secure, but communications problems persist, remove the cable and examine both ends of the cable for bent pins.
2. It is rare (but possible) that the interface port cable located inside the potentiostat may be loose. If this is suspected, disconnect the potentiostat from the power source, remove the cover of the potentiostat, and examine the internal ribbon cables which connect from the back panel down to the main analog circuit board.

AFCBP1 CONTROL MODE

Verify that the potentiostat is turned on and that the Control Mode is set to "external".

1. The "Control Mode" button is located on the lower-left part of the front panel. It must be pushed and held for about one second to change the control mode to "external".

CONCLUSIONS

If all of the above verification steps have been taken, and the AfterMath is still unable to communicate with the CBP bipotentiostat, then the unit may need to be returned to the factory for repair. Contact Pine to arrange a return authorization.