Operating Instructions
for the ASWCV2 PineChem™
Software Package  (version 2.00)

from
Pine Instrument Company
Grove City, Pennsylvania

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Introduction

The PineChem software package is designed for use with the AFCBP1 BIPOTENTIOSTAT manufactured by Pine Instrument Company. The software runs under the Microsoft Windows operating system and utilizes its popular mouse oriented “point-and-click” graphical user interface. Flexible control of the BIPOTENTIOSTAT is provided, allowing the user to perform a wide variety of electrochemical experiments. Once the data from an experiment is acquired and stored in the computer, a complete range of analysis tools are available for graphing and interpreting experimental results.

Scope of this Manual

This manual assumes that the PineChem software package has been successfully installed on your computer system, and that the hardware connection from your computer to the AFCBP1 BIPOTENTIOSTAT is working properly. If this is not the case, then you should refer to the installation instructions which accompany the PineChem software (manual # LMCBP1N).

This manual is written for the professional electrochemist and assumes familiarity with the subject of electrochemistry, especially potential sweep techniques such as cyclic voltammetry. It describes operation of the AFCBP1 BIPOTENTIOSTAT using the PineChem software. Note that the AFCBP1 BIPOTENTIOSTAT can also be operated using its front panel controls. If you are interested in operating the instrument as a stand-alone device, consult the AFCBP1 BIPOTENTIOSTAT Hardware manual.

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Overview

Telling the Software What You Want to Do

In a typical experiment, you first give the software a set of experimental parameters which govern how the experiment is to be conducted. You enter these parameters on an experimental SETUP PANEL, which is a full screen panel that appears on the display whenever you wish to perform a new experiment. Each type of electrochemical experiment has its own unique SETUP PANEL. The parameters on the SETUP PANEL may be entered from the keyboard or loaded from a previously existing file on your hard disk. The latter option lets you perform a new experiment in the same way that you performed an earlier one.

Once the SETUP PANEL is configured properly and you are ready to begin the experiment, the software relays the setup parameters to the BIPOTENTIOSTAT, which, in turn, executes the experiment according to your wishes. The software monitors the progress of the experiment and acquires the experimental data. This data is stored in your computer's memory, and once the experiment is over, it can be graphed, analyzed, and stored on disk for future reference.

How Data is Acquired and Stored

The software acquires data at regular intervals throughout the experiment. Each time a data point is acquired, the potential and current at both working electrodes is recorded and stored in the computer's memory. By the end of the experiment, there is usually a rather large amount of data in memory, and this data can be visualized as a large four-column table of numeric information. Each row in the table consists of four entries referred to as E1, I1, E2, and I2, which are the potentials (E) and currents (I) at electrodes K1 and K2, respectively. Each row of the table is separated in time from the next row by a precise, regular interval of time called the POINT INTERVAL.

Normally, you will want to manipulate, analyze, smooth, and/or graph your experimental results. A variety of software tools are provided to help you do these tasks. It is important to note at the outset, however, that all of these manipulations are carried out on a duplicate copy of the original data. The “raw” experimental data actually acquired during the experiment is referred to as the “ORIGINAL” data set. This ORIGINAL data set is never modified by the software.

Graphing and Analysis

Once the experiment is complete, you may use the software to manipulate a duplicate copy of the experimental data. This duplicate copy is called the “MODIFIED” data set. All of the advanced analysis options, including math and
smoothing functions, operate only on the MODIFIED data set. The ORIGINAL data remains untouched by the software.

Graphing and analysis of the MODIFIED data set is carried out from the ANALYSIS PANEL. Once your experiment is complete, the software automatically displays the ANALYSIS PANEL. A graph of your experimental results occupies most of the space on this panel. A menubar across the top of this window contains numerous display and analysis commands that you can use to manipulate your experimental data.

If at any time during the manipulation of your data, you find that you have hopelessly destroyed the MODIFIED data set (perhaps by smoothing it just a bit too much), you can always go back to the ORIGINAL data set and start over again. This is accomplished by using one of the commands in the Restore menu to copy some or all of the ORIGINAL data set into the MODIFIED data set.

Note that the software also allows you to overlay the ORIGINAL and MODIFIED data sets on the same graph so that you can directly compare them. This is the only overlay option provided by the software.

The software can only work with one set of experimental results at a time. This means that after you have completed an experiment, you should save the experimental data to a disk before proceeding to the next experiment. The software will always warn you if you are about to unintentionally erase unsaved results from a previous experiment or analysis session (see Figure 39).

Between Experiments: The Idle Controls

An important consideration to keep in mind is that the electrochemical cell does not cease to exist between experiments. It is possible (and quite likely) that electrochemical processes will continue to occur in the cell between experiments unless you take some precautions to prevent this from happening. To help you in this task, the software maintains idle control of the BIPOTENTIOSTAT. An INSTRUMENT STATUS panel allows you to set and modify the potentials of the working electrodes before and between experiments. This panel appears when you first start up the software, and it remains available until you exit the software.

Normally, you should choose idle potentials at which no electrochemical process occurs at either working electrode. This helps to keep the solutions in your electrochemical cell unchanged between experiments. Note that the software also allows you to disengage the cell completely between experiments (i.e., the AFCBP1 BIPOTENTIOSTAT can be placed in DUMMY mode).
Performing Experiments

Starting Up the System

In order for the system to function correctly, both the hardware and software must be working together properly. You should always start up the hardware before the software. More details are given below.

Preparing the AFCBP1 BIPOTENTIOSTAT
To place the BIPOTENTIOSTAT under software control, it should be turned on before starting up the software. Once the instrument is turned on, you must switch it from MANUAL to EXTERNAL control by depressing the MODE control button on the front panel of the BIPOTENTIOSTAT. Note that the button must remained depressed for a full second to change the mode.

Starting the PineChem Software Package
The PineChem software can only be activated using the Microsoft Windows operating system. You can start up the package by clicking on the PineChem V2.00 icon located in the PineChem program group. Depending on the speed of your system, the software takes anywhere from 20 to 60 seconds to load into memory.

![Figure 1: The PineChem Program Group](image)

After the software is loaded into memory, the INSTRUMENT STATUS panel appears on the screen. If the system is operating properly, the message on the panel should indicate that the instrument is on-line and ready. The idle controls on the panel are initially set so that the potential applied to each working electrode is zero volts, and the BIPOTENTIOSTAT is placed in DUMMY mode (see Figure 2).

Working without the BIPOTENTIOSTAT
It is possible to run the software in the absence of the AFCBP1 BIPOTENTIOSTAT. If the instrument power is switched off or if it is not placed in EXTERNAL control mode, then the software will notify you that it is unable to communicate with the BIPOTENTIOSTAT. You are given the option to continue without connecting to the instrument. If you choose this option, the software will function normally except that you will not be able to perform experiments. The INSTRUMENT STATUS panel will indicate that the instrument is off-line and unavailable, and the idle controls will be dimmed.
If you wish to operate the software on a computer system that does not contain an INTERFACE BOARD, you should still run the MIO Board Setup utility anyway. This utility is located in the PINECHEM program group (see Figure 1). By simply running the utility and exiting it immediately, a special file called “WDAQCONF.CFG” is created in the WINDOWS subdirectory. This file contains information about which INTERFACE BOARDS (if any) are installed in your system. This file must be present even on systems with no INTERFACE BOARDS.

Multiple INTERFACE BOARDS
In the unusual circumstance that the software recognizes more than one INTERFACE BOARD installed in your system, the software will ask you which board it should use each time you start up the software. This only happens when you have used the MIO Board Setup utility to configure your system for use with more than one compatible INTERFACE BOARD. See the installation instructions for more details regarding multiple boards and/or bipotentiostats.

Problems Loading the Software
If you receive an error message telling you that there is not enough memory to operate the software, try restarting Microsoft Windows and then make certain that the PineChem software is the first application to be activated after the operating system restarts. (This assures that the maximum amount of memory is available to the software when it starts up.) If you still experience memory problems, make sure your system meets the minimum requirements defined in the Installation Manual.

The software may fail to start up properly if any of the files that comprise the software package are missing from your hard drive. If this happens, you should re-install the software package according the instructions in the Installation Manual.

If you forget to place the AFCBP1 BIPOTENTIOSTAT under EXTERNAL control before starting the software, then a warning message appears on the screen, and you are given an opportunity to set up the instrument properly. After you have done this, you may attempt to connect with the BIPOTENTIOSTAT. If a communications error persists, check the cable connections to make certain that they are secure.

Setting up the Idle Conditions
The first action you should take after starting the software is to set up the idle conditions for your working electrodes. Whatever idle settings you choose will take effect immediately. The idle controls on the INSTRUMENT STATUS panel allow you to choose the potentials at which the K1 and K2 electrodes are held in between experiments. Note that these potentials must be specified in millivolts. To change one of these values, simply click inside the appropriate box, change the value, and press the “ENTER” key. You can verify that the proper potential has been applied to the electrode using the front panel voltmeter.
The NORMAL versus DUMMY switch lets you disconnect the electrochemical cell from the BIPOTENTIOSTAT while idling. To do this, simply set this switch to the DUMMY position. This is a good idea if you are working with a particularly sensitive electrochemical cell and you wish to electrically isolate the cell in between experiments. If desired, you can verify the setting of this idle control by observing the front panel indicator on the AFCBP1 BIPOTENTIOSTAT.

![Instrument Status Panel](image)

**Figure 2: The Instrument Status Panel**

Note that you also have the option of placing the BIPOTENTIOSTAT in “open loop” mode while idling. This is essentially the same a placing a high input impedance voltmeter across the working and reference electrodes. This is normally not something you would want to do during a voltammetry session, but might be desirable when working with a pH or ion selective electrode.

Finally, it is important to emphasize that the idle conditions may be adjusted at any time other than during an experiment. The INSTRUMENT STATUS panel can always be made visible by choosing Status... from the Instrument menu that appears on all panels. Making adjustments to the idle conditions is often a good way to test communications between the software and the BIPOTENTIOSTAT.

**Setting Up a New Experiment**

To set up and perform a new experiment, choose the kind of experiment you wish to perform from the Experiment menu. You may choose either the Electrolysis... option or the Analog Sweep Voltammetry... option from this menu. A SETUP PANEL will appear on the screen which contains a variety of controls that you will use to define your experiment. It is important to adjust these controls carefully as they specify the exact parameters that the software uses to perform your experiment.

The appearance of the SETUP PANEL depends on the kind of experiment you choose to perform. The ELECTROLYSIS SETUP PANEL contains all of the controls necessary for an experiment in which the working electrode potentials are held constant throughout the experiment. The ANALOG SWEEP VOLTAMMETRY SETUP
PANEL is a bit larger as it contains controls which let you apply a potential sweep to one or both working electrodes.

Once you have set up the experimental parameters, you may wish to save a copy of the parameters to a setup file on your disk. Once a setup file has been saved to disk, you can just load the parameters from the disk to perform an identical experiment at some point in the future. The Save Setup... and Load Setup... options under the File menu are used to save and load such setup files.
Electrolysis Experiments

The term electrolysis usually refers to techniques where the working electrode potential is held constant throughout the entire experiment. With a BIPOTENTIOSTAT, it is possible to do this with two working electrodes, using the same or two different potentials. To initiate an electrolysis experiment, choose Electrolysis... from the Experiment menu. This causes the ELECTROLYSIS EXPERIMENT SETUP PANEL to appear (see Figure 3).

![Electrolysis Experiment Setup Panel]

**Figure 3: The Electrolysis Experiment Setup Panel**

The controls on this panel are arranged in six groups according to their function. Each of these six groups is discussed in more detail below. There are five buttons along the bottom of the panel and a menubar along the top of the panel.

The LOAD and SAVE buttons are used to read and write the electrolysis setup parameters from or to a hard disk drive. If you will be repeating an experiment many times using the same parameters, it is a good idea to save a copy of the setup to disk for future use.

The DEFAULT button adjusts the settings of all controls on the panel to their default values as shown in Figure 3. These default conditions provide a good starting point for designing new experiments.

Clicking on the PERFORM button initiates the experiment. Clicking on the CANCEL button dismisses the panel without performing the experiment.

The menubar along the top of the panel contains three simple menus. The File menu contains commands that allow you to load and save setup files. You may also exit the software using the Exit command at the bottom of the File menu. The Experiment menu allows you to switch between the various SETUP PANELS for each different kind of experiment.
The **Instrument** menu contains three commands. The **Status...** command activates the **INSTRUMENT STATUS** panel. The **Hardware Devices...** command displays a list of all **INTERFACE BOARDS** and instruments presently connected to the system. The **Reconnect** command can be used to reestablish communications between the software and the instrument when required. The **Instrument** menu is discussed in more detail later.

### Adjusting the Electrolysis Potentials

The controls in the upper left-hand corner of the **SETUP PANEL** are used to specify the initial and electrolysis potentials that are applied to the working electrodes during the experiment. These potentials must be specified in millivolts, and only values between -9995 mV and +9995 mV are permitted.

The **INITIAL POTENTIALS** are imposed on the working electrodes at the onset of the electrolysis experiment. The electrodes remain poised at these initial potentials for a period of time that you specify using the **INITIAL DELAY PERIOD** control. No data is acquired during this initial part of the experiment. The purpose of this brief induction period is simply to provide the cell with some time to come into equilibrium with the initial potentials.

Once the initial delay period has expired, the software begins acquiring data and then, almost immediately, it applies the **ELECTROLYSIS POTENTIALS** to the working electrodes. The sequence in which these two events occurs is important to keep in mind. Data acquisition begins before application of the **ELECTROLYSIS POTENTIALS**. This means that the first few data points are actually acquired before the onset on electrolysis. These first few points are significant, and they play a valuable role in determining the point in time when electrolysis begins.

### When Does Electrolysis Begin?

As long as the initial and electrolysis potentials differ by at least 10 millivolts, there is a noticable jump in the working electrode potential at the onset of electrolysis. After the data acquisition period ends, the software is able to scan through your data and locate this potential jump. This particular location within the acquired data set is then assigned a time value of zero. This provides a time reference that can be used for analysis and graphing.

If a zero time reference is important to you, then you must make certain that there is an observable jump from the initial to the electrolysis potentials of at least 10 millivolts. The software first looks for such a jump in the K1 potential data. If it fails to find a transition in the K1 potential data, then it scans the K2 potential data. If this fails, the software arbitrarily assigns time zero to the first data point acquired.
You can increase the accuracy of the zero time reference by acquiring data using a short POINT INTERVAL. A shorter POINT INTERVAL allows the software to assign time zero to the proper data point with more precision.

**Skipping the Initial Delay Period**

If you wish to skip the initial delay period, simply set the initial and electrolysis potentials to the same value and set the INITIAL DELAY PERIOD control to zero. In this case the software arbitrarily assigns time zero to the first data point acquired.

**Adjusting the Electrolysis Time**

The length of the electrolysis is governed by the EXPT LENGTH control located within the DATA ACQUISITION PERIOD control group on the SETUP PANEL. The numeric control on the left is used to specify the amount of time in terms of whatever time unit you choose using the control on the right (see Figures 4&5).

---

**Figure 4: Adjusting the Electrolysis Time**

You may specify time using any of the following time units: nanoseconds, microseconds, milliseconds, seconds, minutes, hours, days, weeks, or years. If you specify an amount of time that is out of range, the software either adjusts the value so that it is in range or the time control changes to a yellow color to alert you that it is out of range.

**Data Acquisition Timing**

Due to memory limitations and the very nature of digital signal processing, it is impossible for the software to continuously sample the signals from the BIPOTENTIOSTAT in the same fashion as an analog XY recorder would. Rather, the software must sample the incoming signals at regularly spaced intervals. The assumption is that the digitally sampled signal is representative of the actual analog signal. As the sampling frequency is increased, confidence in this assumption also increases. (Unfortunately, so does the need for more memory.)

The reciprocal of the sampling frequency is the POINT INTERVAL. The POINT INTERVAL is amount of time between acquisition of a data point and acquisition of the next data point. Ideally, you would set the POINT INTERVAL to as small a value as possible; however, in most cases this is a needless waste of memory. In general, you just want to take enough data so that a plot of the experimental results has a satisfactory appearance.
The total number of points acquired during an experiment depends on the length of the experiment (EXPT LENGTH) and the POINT INTERVAL. The relationship between these three quantities is given by the expression below:

\[
\text{EXPT LENGTH} = (\text{POINT INTERVAL}) \times (\text{NUMBER OF POINTS})
\]

If any two of these three quantities are known, then the third can be computed easily. For an electrolysis experiment, the software lets you decide which pair of parameters you wish to specify, and then it computes the third automatically. This is accomplished using a set of three radio buttons (see Figure 5) to control which pair is specified.

Clicking on any of the three radio buttons cycles through the three possible pairs of parameters. The default is to specify the length of the experiment (which is the length of the electrolysis) and the number of data points to be acquired. The software then computes the corresponding POINT INTERVAL.

For your reference, the software also computes the sampling rate and an estimated memory requirement. These are not parameters that can be changed directly. The sampling rate is simply the reciprocal of the POINT INTERVAL, and the memory requirement is directly related to the NUMBER OF POINTS.

While you are setting up the data acquisition timing, the software will allow you specify just about anything you desire, no matter how impractical it is. If a particular parameter is out of range, the corresponding control is highlighted using yellow. The software will not allow you to actually perform an experiment until all data acquisition parameters are in range. Table I lists the valid ranges for each of the three parameters.
Table I: Data Acquisition Parameter Summary

<table>
<thead>
<tr>
<th>PARAMETER</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>POINT INTERVAL</td>
<td>Maximum: 655.35 sec (just under 11 minutes)</td>
</tr>
<tr>
<td></td>
<td>Minimum: 42 µsec (for dual electrode experiments)</td>
</tr>
<tr>
<td></td>
<td>22 µsec (for single electrode experiments)</td>
</tr>
<tr>
<td></td>
<td>This parameter is governed by the capabilities of the INTERFACE BOARD.</td>
</tr>
<tr>
<td>NUMBER OF POINTS</td>
<td>Minimum: 5 points</td>
</tr>
<tr>
<td></td>
<td>Maximum: ~200,000*</td>
</tr>
<tr>
<td></td>
<td>The maximum number of points is limited by the amount of memory (RAM and virtual) available on your computer. You should take some time to determine what the practical upper limit is for your system. The number 200,000 is based on a typical 8MB system operating with a significant amount of virtual memory. Note that exceptionally large data sets may slow down the analysis software to a noticeable extent. * Some older INTERFACE BOARDS are limited to 16380 points.</td>
</tr>
<tr>
<td>EXPT LENGTH</td>
<td>Minimum: 110 µsec</td>
</tr>
<tr>
<td></td>
<td>Maximum: ~4 years</td>
</tr>
<tr>
<td></td>
<td>These values computed from minimum and maximum POINT INTERVAL and minimum and maximum NUMBER OF POINTS. During exceptionally long experiments, steps should be taken to prevent loss of power.</td>
</tr>
</tbody>
</table>

Electrode Sensitivities

The term ELECTRODE SENSITIVITY refers to the precision with which current measurements are made at each working electrode. Obviously, you want to make measurements with the greatest degree of precision possible. Given that the BIPOTENTIOSTAT is capable of measuring current levels ranging from milliamps down to nanoamps, the best results are obtained when the BIPOTENTIOSTAT is “tuned” to the proper current range before the experiment is performed.

When using the AFCBP1 BIPOTENTIOSTAT as a stand-alone instrument, this “tuning” process is accomplished by manual adjustment of the front panel CURRENT CONVERTER control. Under software control, however, a working electrode’s sensitivity is adjusted by specifying the maximum current that can be measured at that electrode. If you expect to observe milliamp magnitude currents during your experiment, then you should set the ELECTRODE SENSITIVITY control to one of the milliamp level settings. If your working electrode is very small, and you expect nanoamp level currents, then you should use a nanoamp level setting.

There are twenty-two (22) different current ranges available (see Figure 6). The sensitivity of each working electrode is set independently using “pop-up menu”
controls on the SETUP PANEL. Simply click the mouse on the appropriate control, and then select the desired setting. Alternately, you may use the small arrows on the left side of the control to scroll through all of the possible settings. Note that at the lower current ranges, care must be taken to keep electronic noise from interfering with experimental measurements.

For a given ELECTRODE SENSITIVITY setting, the current is measured with a precision of one part in 4096. For example, if the maximum current is set at 125 mA, the BIPOTENTIOSTAT can measure currents over the range from –125 mA to +125 mA. Dividing this range into 4096 equal-sized increments, you obtain the precision with which current measurements are actually made. In this case, the precision would be 61 microamps.

Obviously, the best precision is obtained when the ELECTRODE SENSITIVITY control is set to as low a current as possible. However, if you set it too low, a large current signal from your experiment might be chopped off, causing some of the peaks in your results to be truncated. On the other hand, if you set it too high, then you needlessly sacrifice measurement precision, and taken to an extreme, the experimental data begins to appear “tiered” like a staircase.

FIGURE 6: SETTING THE ELECTRODE SENSITIVITIES

It is very important to set the ELECTRODE SENSITIVITY correctly. Unfortunately, the only way to do this is to know in advance the magnitude of the highest current that will be observed during the experiment. Unless you have already performed a similar experiment or are capable of predicting the future, you will have to set this control by trial-and-error by repeating the experiment more than one time.

Automated Range Checking

The software has a feature which can assist you in choosing the proper ELECTRODE SENSITIVITY. If you turn on either or both of the POST-EXPERIMENT RANGE CHECKING options located immediately under the ELECTRODE SENSITIVITY

Check here if you want the software to verify that you used the correct current range after the experiment concludes

Choose the current range that most closely matches the current you expect to observe during the experiment

Electrode Sensitivities

K1 Current (max) 1 A
K1 post-experiment range check
K2 Current (max) 1 A
K2 post-experiment range check

1 A
500 mA
250 mA
100 mA
... etc ...
1000 μA
500 μA
250 μA
... etc ...
250 nA
100 nA
controls (see Figure 6), then at the conclusion of the experiment, the software automatically analyzes your experimental results to see if better settings could have been used. If so, you are given the option of repeating the experiment again using the better settings.

Note that this range checking occurs after the experiment has concluded. You must actually perform the experiment once in order for the software to be able to predict the best current range setting.

**Delay Sequences**

At the very beginning of an experiment, initial potentials are applied to both working electrodes. The next major event is the application of the electrolysis potentials. In most cases, it is a good idea to put a delay between the application of initial potentials and electrolysis potentials. During this so-called *induction period*, the solution has time to come to equilibrium with the initial potentials applied to the electrodes.

Figure 7 shows the group of SETUP PANEL controls which allow you to adjust the length of this initial delay period and that for a final delay period as well. The default delay times are three seconds, but you can change these to any value between zero and 300 seconds (5 minutes).

![Delay Periods Table]

**Figure 7: Delay Times for Induction and Relaxation Periods**

Similarly, at the end of the experiment (after the data acquisition period has expired) you may wish to hold the electrolysis potential for an additional brief period of time before applying the idle conditions to the electrodes. You can specify the length of this *relaxation period* using the other FINAL DELAY control. In most cases there is no need for a relaxation period, but again, any value between zero and 300 seconds may be specified.

It is important to keep in mind that data is not acquired during the induction period or the relaxation period. (Actually, a few data points from the induction period are acquired, as discussed earlier, so that the software can observe the onset of electrolysis.) Long delay times are not recommended as you do not regain control of the software until they expire.

**Post-Experiment Idle Conditions**

Once the electrolysis ends and after the relaxation period (if any) expires, the experiment is concluded, and a set of idle conditions is imposed on the electrochemical cell to preserve it for the next experiment.
A set of controls in the lower right-hand part of the SETUP PANEL allow you to specify these post-experiment idle conditions (see Figure 8). Notice that these idle controls have the same appearance and function as those found on the INSTRUMENT STATUS panel. Once the experiment concludes, the INSTRUMENT STATUS panel is updated to reflect any changes in the idle conditions.

**A Caveat Regarding Idle Conditions**

It is an easy matter to specify post-experiment idle potentials that differ significantly from the electrolysis potentials applied during the experiment. Keep in mind, however, that the sudden imposition of the idle potentials will most likely be accompanied by short-lived, but very large, current transients. Sensitive electrochemical systems may be damaged by such transients, and when working with these systems, care should be taken to adjust the idle potentials so that they match the initial and/or electrolysis potentials.

**Miscellaneous Options**

Electrolysis Using a Single Working Electrode

While the principle strength of a BIPOTENTIOSTAT is its ability to work with two independent working electrodes, it is, of course, possible to use it with a more traditional electrochemical cell that has only one working electrode. To perform an electrolysis experiment using just one working electrode, it is recommended that you connect the working electrode to the K1 electrode circuit. No connection to the K2 electrode should be made, and the potential applied to the K2 circuit should be held constant at zero volts.

In addition, you should check the IGNORE K2 ELECTRODE option on the SETUP PANEL (see Figure 9). When this option is checked, the software does not acquire data from the second working electrode (K2). This saves both time and memory. With this option checked, it is possible to acquire data more rapidly.
(every 22 microseconds) because only the E1 and I1 signals are monitored. Less memory is required, allowing you to acquire a larger number of data points.

**Using the Dummy Cell**

If you wish to test your experimental parameters without actually performing a “real” experiment, you can check the PERFORM USING DUMMY CELL option (see Figure 9). With this option enabled, the electrolysis experiment is performed using the internal DUMMY resistor network in the BIPOTENTIOSTAT. The actual electrochemical cell remains disengaged throughout the experiment.

**Using an Externally Generated Potential Waveform**

If you wish, you can acquire data from an electrochemical experiment where the potential waveform comes from some external source such as a pulse or function generator. Several steps are recommended if you will be performing this kind of experiment.

First, the signal output(s) from the function generator must be connected to the AFCBP1 BIPOTENTIOSTAT. Both of the electrode circuits (K1 and K2) have independent signal inputs on the front panel of the BIPOTENTIOSTAT. These inputs are labeled “IN” on the front panel. Any voltage placed on these inputs is summed with the output of the analog SWEEP GENERATOR (if active) and any potential offset applied by the DAC circuitry on the INTERFACE BOARD. The sum is then applied to the appropriate working electrode.

Second, you should use the ELECTROLYSIS SETUP PANEL to prepare the software for data acquisition. You will have to set the POINT INTERVAL, NUMBER OF POINTS, and/or EXPT LENGTH to match whatever external signal you are applying to the cell. Make sure that the EXPT LENGTH is long enough to acquire data throughout the entire experiment, and be sure to include some extra time at the start of the experiment during which you can manually initiate the external waveform sequence. (No hardware triggering mechanism is available using the present release of this software.)

Third, on the ELECTROLYSIS SETUP PANEL, you should enter zero millivolts for the INITIAL POTENTIALS and the ELECTROLYSIS POTENTIALS. Using non-zero potentials will cause your external potential waveform to be offset by whatever amount you enter into these controls. This is usually not desirable.

Finally, you should check the USE 10V INPUT RANGE option on the SETUP PANEL (see Figure 9). This disables the software’s automatic potential range selection feature and forces the software to acquire potential signals using the full ±10 volt input range. (When this option is not active, the software automatically chooses which input range most closely matches the potentials specified on the SETUP PANEL. The available ranges are ±10V, ±5V, and ±2.5V.)
Executing the Experiment

Once you have made all of the adjustments to the SETUP PANEL controls, you are ready to begin execution of the experiment. Before starting the experiment, you may wish to save your setup to disk using the Save Setup... option in the File menu. Once saved, you can recall the settings at a future date if you ever wish to perform a similar experiment.

To initiate the experiment, click on the PERFORM button. This dismisses the SETUP PANEL and switches to the ELECTROLYSIS IN PROGRESS window. If the software is unable to perform the experiment for any reason, you will receive an error message and be returned to the SETUP PANEL.

The ELECTROLYSIS IN PROGRESS window (see Figure 10) lets you watch what is going on during your experiment. At the start of the experiment, the INITIAL POTENTIALS are applied to both working electrodes, and the induction period begins. A timer counts down the seconds remaining in the induction period.

![ELECTROLYSIS IN PROGRESS window](image)

**Figure 10: The Electrolysis in Progress Window**

Near the very end of the induction period, data acquisition commences and then the ELECTROLYSIS POTENTIALS are applied. Several indicators let you keep up with the progress of the experiment. A large graph at the top of the panel usually lets you observe the experimental results in real time. The only exception is when the experiment ends so soon that there isn’t enough time to
plot the results as they are acquired. The graph shows a plot of current versus time for one or both working electrodes.

Once the experiment concludes, the relaxation period begins. You can observe the seconds counting down during this period. After the relaxation period, the POST-EXPERIMENT IDLE CONDITIONS are imposed on the electrochemical cell, and in most cases, the software switches to the ANALYSIS PANEL to let you observe your results.

**Post-Experiment Range Checking**

If you have switched on one or both of the AUTOMATIC RANGE CHECKING features, the software examines current signal(s) just acquired to make sure that you used reasonable ELECTRODE SENSITIVITY settings. If the software detects that you could have used a better setting, a message appears on the screen.

In the lower-left corner of this message panel (see Figure 11), the software indicates the largest observed current at each working electrode. In the example shown, the largest K1 working electrode current was 500 µA. It is quite likely that even larger currents were passed at the K1 working electrode, but because the K1 ELECTRODE SENSITIVITY was set at 500 µA, these larger currents were too high to be observed. The proper course of action in this case is to REPEAT the experiment with the K1 ELECTRODE SENSITIVITY set to higher current range—perhaps the 2.5 mA range.

![Figure 11: The Post-Experiment Range Checking Feature](image)

The range checking feature can also detect if you have set the ELECTRODE SENSITIVITY to a current range that is much higher than necessary. For example, if you set the ELECTRODE SENSITIVITY to the 5 mA range, but the largest observed current is only 1.3 µA, then the software will give you the chance to repeat the experiment again using a smaller current range—perhaps the 2.5 µA range.
To repeat the experiment using better ELECTRODE SENSITIVITIES, simply adjust the current ranges displayed on the RANGE CHECKING panel. Then, click on the REPEAT button to try again.

If you wish to retain the data from the experiment, click on the KEEP DATA button, and the software switches to the main ANALYSIS PANEL to let you observe and work with your results.

To return to the SETUP PANEL without repeating the experiment or retaining any acquired data, click on the CANCEL button.

**Halting an Experiment in Progress**

The STOP button on the ELECTROLYSIS IN PROGRESS window lets you stop an experiment in progress. This is useful when you see that things aren’t going well on the “real time” display. If some data has already been acquired at the time you stop the experiment, the software switches to the ANALYSIS PANEL, and the (incomplete) data set is made available for graphing and analysis.
Analog Sweep Experiments

A variety of electrochemical techniques that require a smooth, analog potential sweep to be applied to one or both working electrodes can be designed using the generic ANALOG SWEEP EXPERIMENT SETUP PANEL. Such techniques include cyclic voltammetry, rotated disk and ring-disk voltammetry, and anodic stripping voltammetry. To initiate a sweep experiment, choose Analog Sweep Voltammetry... from the Experiment menu. This causes the ANALOG SWEEP EXPERIMENT SETUP PANEL to appear (see Figure 12).

![Figure 12: The Analog Sweep Experiment Setup Panel](image)

The controls on this panel are arranged in six groups according to their function. Each of these groups is discussed in more detail below. There are five buttons along the bottom of the panel and a menubar along the top of the panel.

The LOAD and SAVE buttons are used to read and write the analog sweep experiment setup parameters from or to a hard disk drive. If you will be repeating an experiment many times using the same parameters, it is a good idea to save a copy of the setup to disk for future use.

The DEFAULT button adjusts the settings of all controls on the panel to their default values. These default conditions provide a good starting point for designing new experiments.

Clicking on the PERFORM button initiates the experiment. Clicking on the CANCEL button dismisses the panel without performing the experiment.
The menubar along the top of the SETUP PANEL contains three simple menus. The **File** menu contains commands that allow you to load and save setup files. You may also exit the software using the **Exit** command at the bottom of the **File** menu. The **Experiment** menu allows you to switch between the various SETUP PANELS for each different kind of experiment.

The **Instrument** menu contains three commands. The **Status...** command activates the INSTRUMENT STATUS panel. The **Hardware Devices...** command displays a list of all INTERFACE BOARDS and instruments presently connected to the system. The **Reconnect** command can be used to reestablish communications between the software and the instrument when required. The **Instrument** menu is discussed in more detail later.

### Dual Electrode Sweep Methods

With a Bipotentiostat, it is possible to apply the analog sweep to one or both working electrodes. This leads to three different possibilities depending on whether the sweep is applied to K1, K2, or both K1 and K2. The terms **collection**, **shielding**, and **window** have come to be associated with these three possibilities. These terms, which are summarized in Table II, find their origins in the lore of hydrodynamic voltammetry using a rotated ring-disk electrode (RRDE).

The terms **collection** and **shielding** refer to the two traditional modes of operation for an RRDE. Both terms refer to experiments where one electrode potential is swept while the other is held constant. In a **collection** experiment, the K1 (disk) potential is swept while the K2 (ring) potential is held constant. The roles are reversed in a **shielding** experiment. (When using an RRDE, it is assumed that the disk and ring are connected to K1 and K2, respectively. In other dual working electrode experiments, you may connect the electrodes in any manner you like.)

The term **window** is used to label what has traditionally be called a “scanning window” or “moving window” experiment. This is an experiment in which a potential sweep is applied to both working electrodes. Identical sweeps are applied to both working electrodes, but one waveform is usually offset from the other by some constant potential difference.

![Image](image.png)

**Figure 13: Choosing the Type of Sweep Experiment**

To choose between the three possible variants, use the **EXPERIMENT TYPE** control located in the upper left-hand corner of the SETUP PANEL. This is a pop-up menu control (see Figure 13) that is operated by clicking the mouse.
anywhere on the control. The function of some of the other controls on the SETUP PANEL is altered to match your choice of experiment type. Thus, it is very important to choose the type of experiment first, before any other controls are used.

Table II: The Three Types of Sweep Experiments

<table>
<thead>
<tr>
<th>EXPT TYPE</th>
<th>DESCRIPTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>COLLECTION</td>
<td>The potential of the K1 working electrode is swept. The potential of the K2 working electrode is held constant. The term <em>collection</em> is derived from the lore of rotated ring-disk voltammetry, where the potential of the disk electrode (K1) is swept while the potential of the ring electrode (K2) is held constant. This type of experiment has other uses outside the context of a rotated ring-disk electrode (RRDE).</td>
</tr>
<tr>
<td>SHIELDING</td>
<td>The potential of the K1 working electrode is held constant. The potential of the K2 working electrode is swept. Again, the term <em>shielding</em> is derived from the RRDE literature.</td>
</tr>
<tr>
<td>WINDOW</td>
<td>The potentials of both the K1 and K2 electrodes are swept. The term <em>window</em> is a shortened form of the term <em>scanning window</em> that is used to describe an experiment where both potentials are swept with a fixed potential offset between them.</td>
</tr>
</tbody>
</table>

Single Electrode Sweep Methods

While the principle strength of a BIPOTENTIOSTAT is its ability to work with two independent working electrodes, it is, of course, possible to use it with a more traditional electrochemical cell that has only one working electrode. To perform a sweep experiment using just one working electrode, it is recommended that you connect the working electrode to the K1 electrode circuit and perform a *collection* experiment. No connection to the K2 electrode should be made, and the potential applied to the K2 circuit should be held constant at zero volts. Any bogus data acquired from the K2 electrode circuit can be ignored.

Defining the Sweep Program

A detailed discussion of the controls on the SETUP PANEL which govern the sweep program follows. For the most part, the controls are shared in common by *collection*, *shielding*, and *window* experiments. The labels on the controls are changed slightly depending on the type of sweep experiment being performed. Because of the complex way in which these controls sometimes interact, the SWEEP DISPLAY GRAPH in the upper right-hand corner of the SETUP PANEL is constantly updated to give you visual feedback regarding the sweep program you have chosen. This graph is discussed in more detail later on.
Choosing the Sweep Rate
No matter how complex a sweep program you design, the same sweep rate is used throughout the experiment. This sweep rate is set using the SWEEP RATE control (see Figure 14). To set the sweep rate, simply click the mouse inside the text box and type in any desired sweep rate from 5 to 9,995 mV/s. The sweep rate must be a multiple of 5 mV/s, and the software automatically rounds your choice to the nearest multiple. A sweep rate of zero is not permitted. Minor adjustments in the sweep rate can be made by clicking on the two small arrow controls just to the left of the text box.

You may enter any sweep rate between 5 and 9995 mV/sec using 5 mV/sec increments.

The number of sweep segments must be between 1 and 60000.

The direction for the first sweep segment is specified here.

Selecting the Initial Sweep Direction
The INITIAL DIRECTION control is a switch which determines the initial direction of the potential sweep. The UP direction corresponds to a sweep which goes in the positive (anodic) direction. The DOWN direction corresponds to a sweep which initially heads in the negative (cathodic) direction.

Setting the Number of Sweep Segments
A “sweep segment” is simply a portion of the overall sweep program during which the potential moves in the same direction from one limit potential to another limit potential. The entire sweep program is made up of one or more of these sweep segments. Typically, anywhere from one to three sweep segments might be used, but you may specify any number of sweep segments up to 60000 (provided your computer has enough memory).

The simplest sweep program is that used for linear sweep voltammetry. In this case, the sweep program consists of just one segment. The sweep starts at one limit potential and ends at some other limit potential. The single sweep is either anodic or cathodic depending on the setting of the INITIAL DIRECTION switch. When two or more sweep segments are used, the experiment is usually called cyclic voltammetry.

Choosing the Sweep Limit Potentials
You always have to specify at least two limit potentials to define a potential sweep program. In general, these two potentials define the upper and lower boundary potentials for each sweep segment. The simplest sweep programs are those which are defined in terms of just these two limit potentials. More complex
sweep programs are possible if you decide to use a unique initial potential and/or a unique final potential.

Figure 15 shows the sweep limit controls for a simple sweep program. The particular case shown is from a collection experiment, so the controls are labeled “K1 Sweep Potentials” to indicate that the sweep is being applied to the K1 working electrode. Also, this particular sweep program has an even number of sweep segments (six), so the initial and final potentials are the same.

To start the first sweep segment at a unique potential, activate this radio button and enter the desired potential in the box.

To stop the last sweep segment at a unique potential, activate this radio button and enter the desired potential in the box.

The two LIMIT POTENTIALS should be entered into these boxes. The lower potential goes in the left-hand box, and the upper goes in the right-hand box.

These two boxes normally indicate the default initial and final potentials. To change these values, you must activate the corresponding radio button and then enter the new (unique) value in the box.

**FIGURE 15: THE SWEEP LIMIT CONTROLS**

**Simple Sweep Programs**

A simple sweep program is one in which every sweep segment in the sweep program is bounded by the same two limit potentials. This kind of sweep program is often used in long-running multiple sweep experiments like cyclic voltammetry. For this simple case, you need only enter the two limit potentials into the two LIMITS boxes. The lower limit should be entered in the left-hand box and the upper limit should be entered into the right-hand box.

You do not have to specify the initial potential or the final potential. The software already knows which limit potential should be the initial potential based on the direction of the first sweep segment. For example, if the initial sweep direction is down (cathodic), then the higher limit potential becomes the initial potential. Similar logic is used to determine the final potential. A more complete discussion of this logic is given later (see “More About the Initial Potential and the First Sweep Segment” and “More About the Final Potential and the Last Sweep Segment”, below).

**Which Working Electrode Potential is Swept?**

Look closely at Figure 16 and note how the K1 and K2 designations switch places depending on which type of experiment is being performed. (The colors of the corresponding controls are also switched.) When you use the EXPERIMENT TYPE control (see Figure 13) to switch between collection and
what about the other electrode?
control of the “other” electrode potential is quite straightforward. for both collection and shielding experiments, the other electrode is simply held at a constant potential. the text box labeled hold at is where you specify the potential at which the other electrode is to be held (see figure 16). if you are not using a second working electrode, then you should set this control to zero millivolts.

sweep programs with unique initial and final potentials
if you need to use a unique initial potential or a unique final potential, you must activate the appropriate radio button as described in figure 13. once you have activated one or both of these buttons, the sweep limit controls on the setup panel will look more like those depicted in figure 17. the text boxes for the initial and/or final potentials are active and can be set to values other than the default potentials.

in the particular example in figure 17, both the initial and final potentials are unique. the first sweep segment starts at 300 mV, and the last sweep segment has been adjusted to end at 600 mV. note that all of the sweep segments in the “middle” are bounded by the limits 800 mV and 0 mV. (the K2 potential is held constant at 100 mV throughout the experiment.)

the software automatically keeps track of the direction (i.e., up or down) for each sweep segment in the sweep program. the direction is based on your choice for the initial direction. the software uses this information to determine which of the two limit potentials is the appropriate “turn-around” point for a particular sweep segment. during the actual experiment, the analog sweep generator inside the AFCBP1 Bipotentiostat reverses the sweep direction at these “turn-around” points.
More About the Initial Potential and the First Sweep Segment

The very first sweep segment obviously starts at the initial potential. For a simple sweep program, the software chooses one of the two limit potentials to be the initial potential. The choice is rather clear. If the initial sweep direction is up, then the initial potential defaults to the lesser of the two limit potentials. Conversely, if the initial sweep direction is down, then the greater limit potential is the default.

When you choose to specify a unique initial potential, you are essentially overriding the default. The software does, however, check to make sure your choice makes sense. That is, it makes sure your choice of initial potential and initial direction does, in fact, produce sweep segment that will eventually encounter a limit potential rather than head off forever in the wrong direction. If your choice does not make sense, then the software substitutes the default.

More About the Sweep Segments “in the Middle”

Any sweep program with more than two sweep segments is going to have at least one sweep segment that can be described as being “in the middle” of the sweep program. The “middle” part of a sweep program is characterized by repetitive sweeping between two limit potentials. All of these “middle” sweep segments share a few things in common. They start at one limit potential and end at the other limit potential. They are all the same length. They are also unaffected by your choice of unique initial and/or final potentials.

More About the Final Potential and the Last Sweep Segment

The direction of the final sweep is determined from the initial sweep direction and whether the total number of sweep segments is an even or odd number. The last sweep segment obviously ends at the final potential. For a simple sweep program, the software chooses one of the two limit potentials to be the final potential. Again, the choice is rather clear. If the last sweep segment is sweeping up, then the final potential defaults to the higher of the two limit potentials.
potentials. Conversely, if the last sweep segment is sweeping down, then the lesser limit potential is the default.

When you choose to specify a unique final potential, you are overriding the software’s default choice. If you specify a unique final potential, it must be a potential that makes sense. That is, it must be a potential that falls somewhere along the path taken by the final sweep. If it is not, the software automatically substitutes the default instead of your choice.

Scanning Window Experiments
If you wish to apply a potential sweep program to both working electrodes at the same time, you should set the EXPERIMENT TYPE control to the window setting. This configures the software so that the potential sweep applied to the K1 working electrode is also applied to the K2 electrode. An additional offset potential may be specified for the K2 electrode so that there is a fixed potential difference between the two working electrodes at all times during the experiment.

When you are designing a window experiment, you should first define the sweep program in terms of the K1 electrode just as you would for a collection experiment. Then, decide what offset potential to use for the K2 electrode and enter it into the OFFSET BY text box.

The Sweep Display Graph
In the upper right-hand corner of the SETUP PANEL, there is a large graph which displays the potentials that are applied to the working electrodes during an experiment. A considerable amount of information is placed on this graph, and it is updated constantly as you adjust the various experimental parameters on the SETUP PANEL. A quick glance at this graph lets you see all of the key aspects of a particular experiment.

There are always two traces displayed on the graph. One trace is plotted in yellow while the other is plotted in blue. The yellow trace represents the potential applied to the K1 electrode, while the blue trace represents the K2 electrode.

The initial and final delay periods are displayed as horizontal line segments on the extreme left and right hand sides of the graph. If the duration of a delay period is zero, then it does not appear on the graph. The SWEEP DISPLAY GRAPH in Figure 17 shows both delay periods. Note that the potentials imposed on the electrodes during the final delay period are the same as the initial potentials.

There are three important aspects of an experiment that are not reflected on the SWEEP DISPLAY GRAPH. First, no indication is given as to whether the potentials are ever actually applied to the electrode. That is, there is no way to tell from the graph if the BIPOTENTIOSTAT is in DUMMY mode before, during, or after the
experiment. Second, the idle potentials before and after the experiment are not present on the graph. Third, because of space constraints, no more than twenty sweep segments are plotted on the graph at the same time. Sweep programs with more than twenty sweep segments are graphed in an abbreviated fashion.

Delay Sequences
At the very beginning of an experiment, initial potentials are applied to both working electrodes. The next major event is the start of the potential sweep program. In most cases, it is a good idea to insert a time delay between the application of initial potentials and the start of the sweep. This time delay provides an “induction period” during which the electrodes can come into equilibrium with the surrounding solution. During this induction period, both electrodes are held at their initial potentials for a few seconds, and then the sweep program begins.

Figure 7 shows the SETUP PANEL control which allows you to adjust the length of this time delay before the onset of the potential sweep program. The three second delay shown is quite sufficient in most cases; however, you may specify any value between zero and 300 seconds (5 minutes). Usually you will want to specify the time delay in seconds, but if you wish, the time unit can be changed.

Similarly, at the end of the experiment (after the potential sweep program) you may wish to pause before applying the idle conditions to the electrodes. You can specify the length of this “relaxation period” using the FINAL DELAY control. The default setting for this control is zero, but again, any value up to 300 seconds may be specified.

It is important to keep in mind that data acquisition only occurs during the sweep program. No data is acquired during either delay period. Long delay times are not recommended as you do not regain control of the software until they expire.

A Caveat Regarding the Final Delay Period
During the relaxation period, the working electrodes are held at the initial potentials. For a simple sweep program with an even number of sweep segments, the final potentials and initial potentials are the same. However, sweep programs with an odd number of sweep segments or those with a unique final potential will generally end at a potential that is quite different than the initial potential. At the end of the sweep program, the working electrode potentials are suddenly changed back to the initial potentials and remain there for the duration of the relaxation period. This sudden transition may harm sensitive electrochemical systems, and care should be taken to avoid this condition when working with such systems.

Data Acquisition
During an analog sweep voltammetry experiment, the software monitors the current and potential at both working electrodes, thus, a total of four signals are
acquired. The software samples the incoming signals at a regular time interval known as the POINT INTERVAL. (The reciprocal of the POINT INTERVAL is the sampling frequency.) Ideally, you would set the POINT INTERVAL to as small a value as possible; however, in most cases this is a needless waste of memory. In general, you just want to take enough data so that a plot of the experimental results has a satisfactory appearance.

The NUMBER OF POINTS acquired during an experiment depends on the length of the experiment (EXPT LENGTH) and the POINT INTERVAL. The relationship between these three quantities is given by the expression below:

\[
\text{EXPT LENGTH} = (\text{POINT INTERVAL}) \times (\text{NUMBER OF POINTS})
\]

In a sweep experiment, the EXPT LENGTH is determined by the sweep program. For a given set of sweep program parameters, the EXPT LENGTH is a fixed and known quantity that the software computes for you automatically.

In order for the software to know how often to sample the signals from the BIPOTENTIOSTAT, you must specify either the POINT INTERVAL or the NUMBER OF POINTS. (Given one of these two quantities, the software can compute the other.) The default condition is for you to specify the POINT INTERVAL and let the software compute the NUMBER OF POINTS for you. You may configure the software for the other case if you wish.

![Data Acquisition Period](image)

**Figure 18: The Sweep Experiment Acquisition Timing Controls**

Keep in mind that all data acquisition parameters are subject to hardware and memory constraints as described in Table I. You can enter just about any parameters you wish, but the software will not permit you to actually perform an experiment until the parameters are within the proper range.
Setting the POINT INTERVAL to a Fixed Time Increment

If you prefer to specify the POINT INTERVAL in terms of time, then the controls in the DATA ACQUISITION section of the SETUP PANEL should be configured as shown in Figure 18. You can do this by clicking on the radio button next to the POINT INTERVAL control until it is activated.

The POINT INTERVAL is set by entering the amount of time into the text box and choosing the appropriate time units using the pop-up menu to the right of the text box. The example in Figure 18 shows a POINT INTERVAL equal to 500 microseconds for an experiment where the sweep program duration is 5 seconds. Note how the software computes that 10000 points are to be acquired during this experiment. You may set the POINT INTERVAL to any amount of time that you like, subject to the limitations presented in Table I.

Setting the POINT INTERVAL to a Fixed Potential Increment

You may find it easier to think about the POINT INTERVAL in terms of potential rather than time because voltammograms are generally plotted as current vs. potential rather than current vs. time. By setting the POINT INTERVAL unit control to mV (see Figure 19), you may specify the interval in terms of millivolts. The software will internally converts the potential increment to a corresponding time interval using the SWEEP RATE.

If you plan on repeating the same experiment, each time using a different SWEEP RATE, then it is a good idea to specify the POINT INTERVAL in terms of millivolts. This assures that the data sets from all of the experiments will have the same total NUMBER OF POINTS and the same general appearance when plotted.

Data is to be acquired every time the sweep output moves 50 mV. Given that the SWEEP RATE is 400 mV/sec in this example, the POINT INTERVAL (in terms of time) is actually 125 msec.

FIGURE 19: SETTING THE POINT INTERVAL USING A POTENTIAL

Setting the NUMBER OF POINTS

If you prefer not to think about the POINT INTERVAL at all, then you have the option of specifying the NUMBER OF POINTS to be acquired during the sweep program (see Figure 20). In this case, the software computes the POINT INTERVAL for you.
The software computes the data acquisition rate and required memory and puts this information on the SETUP PANEL. The acquisition rate is the reciprocal of the POINT INTERVAL. The required memory is an estimate based on the NUMBER OF POINTS.

Electrode Sensitivities
The term ELECTRODE SENSITIVITY refers to the precision with which current is measured during the experiment. The sensitivity controls on the ANALOG SWEEP VOLTMETRY SETUP PANEL are identical to those found on the ELECTROLYSIS EXPERIMENT SETUP PANEL. For more details, refer back to Figures 6 & 11 and the related discussion.

Post-Experiment Idle Conditions
Once the relaxation period (if any) expires, the experiment is concluded, and a set of idle conditions is imposed on the electrochemical cell to preserve it for the next experiment.

FIGURE 21: POST-EXPERIMENT IDLE CONDITIONS
A set of controls in the lower right-hand part of the SETUP PANEL allow you to specify these post-experiment idle conditions (see Figure 21). Notice that these idle controls have the same appearance and function as those found on the
INSTRUMENT STATUS panel. Once the experiment concludes, the INSTRUMENT STATUS panel is updated to reflect any changes in the idle conditions.

A Caveat Regarding Idle Conditions
It is an easy matter to specify post-experiment idle potentials that differ significantly from the potentials applied during the experiment. Keep in mind, however, that the sudden imposition of the idle potentials will most likely be accompanied by short-lived, but very large, current transients. Sensitive electrochemical systems may be damaged by such transients, and when working with these systems, care should be taken to adjust the idle potentials so that they match the initial and/or final potentials.

Miscellaneous Options
The OTHER OPTIONS section of the ANALOG SWEEP EXPERIMENT SETUP PANEL (see Figure 22) is very similar to that for the ELECTROLYSIS EXPERIMENT SETUP PANEL. One difference is that you cannot disable the K2 electrode during an analog sweep experiment.

![Figure 22: Miscellaneous Setup Panel Options]

- Perform Using Dummy Cell
- Use 10 V Potential Input Range
- Ignore K2 Electrode

Using the Dummy Cell
If you wish to test your sweep program without actually performing a “real” experiment, you can check the PERFORM USING DUMMY CELL option. With this option enabled (see Figure 22), the sweep experiment is performed using the internal “dummy” resistor network within the BIPOTENTIOSTAT. The actual electrochemical cell remains disengaged throughout the experiment.

Using an Externally Generated Potential Waveform
When working with an externally generated waveform, it is recommended that you use the ELECTROLYSIS EXPERIMENT SETUP PANEL to configure the software for data acquisition. This is discussed in detail in an earlier section of this manual.

Although it is not recommended, if you intend to apply an externally generated potential signal to the working electrode(s) during an analog sweep experiment, you should probably check the USE 10V INPUT RANGE option on the SETUP PANEL (see Figure 22). This disables the software’s automatic potential range selection feature and forces the software to acquire potential signals using the full ±10 volt input range. Keep in mind that the externally supplied signal is summed with the SWEEP GENERATOR output, and it is this sum which is applied to the working electrode(s).
Executing the Experiment

Once you have made all of the adjustments to the SETUP PANEL controls, you are ready to begin execution of the experiment. Before starting the experiment, you may wish to save your setup to disk using the Save Setup... option in the File menu. Once saved, you can recall the settings at a future date if you ever wish to perform a similar experiment.

To initiate the experiment, simply click on the PERFORM button on the SETUP PANEL. This dismisses the SETUP PANEL and switches to the ANALOG SWEEP IN PROGRESS window. If the software is unable to perform the experiment for any reason, you will receive an error message and be returned to the SETUP PANEL.

The ANALOG SWEEP IN PROGRESS panel lets you watch what is going on during your experiment. The appearance of this panel is very similar to that used during an electrolysis experiment (see Figure 10). At the start of the experiment, the INITIAL POTENTIALS are applied to both working electrodes, and the induction period begins. A timer counts down the seconds remaining in the induction period.

Data acquisition begins as soon as the sweep program starts. Several indicators let you keep up with the progress of the experiment. A large graph at the top of the panel usually lets you observe the experimental results in real time. The only exception is when the experiment ends so soon that there isn’t enough time to plot the results as they are acquired. The graph shows a plot of current versus time for both working electrodes.

Once the experiment concludes, the relaxation period begins. You can observe the seconds counting down during this period. After the relaxation period, the POST-EXPERIMENT IDLE CONDITIONS are imposed on the electrochemical cell, and in most cases, the software switches to the ANALYSIS PANEL to let you observe your results.

Post-Experiment Range Checking
If you have switched on one of the AUTOMATIC RANGE CHECKING features for the electrode sensitivities, the software performs a post-experiment check of your results to make sure you used the best possible maximum current settings. If it detects that you could have used a better setting, a message panel appears on the screen. See Figure 11 and the related discussion for more details regarding this feature.

Halting an Experiment in Progress
The STOP button on the EXPERIMENT IN PROGRESS window lets you halt an experiment in progress. This is useful when you see that things aren’t going well on the “real time” display. If some data has already been acquired at the time you stop the experiment, the software switches to the ANALYSIS PANEL, and the (incomplete) data set is made available for graphing and analysis.
Figure 23: The Main ANALYSIS PANEL
Viewing the Results

Once an experiment has been successfully completed, there is usually a large amount of data in your computer’s memory ready to be graphed and analyzed. The ANALYSIS PANEL, which normally appears at the end of an experiment, features a large graph of the acquired signals (see Figure 23). All data analysis and graphing options are available from this panel. Keep in mind that even while working with your experimental results, the software maintains control of the BIPOTENTIOSTAT. This means you can make adjustments to the idle controls on the INSTRUMENT STATUS panel even while working with your results.

The Analysis Graph

Most of the area on the ANALYSIS PANEL is reserved for graphing data. The menus across the top of the panel (described in the next section) contain most of the analysis commands. Along the side of the panel are a variety of buttons and text boxes used to manipulate the graph and to display quantitative information. The use of these buttons and the on-screen cursors is described in more detail below. The main features on the graph are an orange trace corresponding to the K1 electrode data and/or a purple trace corresponding to the K2 electrode data.

The units on the X and Y axes appear in the title above the graph. Depending on how you are viewing your data, the X axis units will either be time or millivolts. When you are viewing your data as current versus potential (i vs. E), the X axis units are in millivolts. Cathodic (more negative) potentials are plotted toward the left, and anodic (more positive) potentials are plotted toward the right. Note that this is the European rather than the American convention. The potential is indicated at several tick marks along the X axis. When you are viewing your data as current vs. time (i vs. t) or potential vs. time (E vs. t), the X axis units will be some appropriate unit of time.

The Y axis units vary significantly depending on just what is being displayed. Experimental results from a voltammetry experiment are typically plotted as a graph of current vs. potential (i vs. E) or current versus time (i vs. t). In either of these two cases, the Y axis units on the graph are usually amps, milliamps, or microamps, according to the level of current actually observed during the experiment. On graphs displaying the current signals from both working electrodes, the same scale is used to plot both signals.

Cathodic (reduction) currents are plotted toward the bottom of the graph while anodic (oxidation) currents are plotted toward the top of the graph. The graph includes up to eight Y axis tick mark labels that are usually rounded to the nearest multiple of 2, 5, or 10.
If you choose to view the potential sweep data (E vs. t), then the Y axis units are volts or millivolts, with cathodic (more negative) potentials plotted toward the bottom of the graph, and anodic (more positive) potentials plotted toward the top of the graph. This type of graph is useful for viewing the actual potential signal applied to the working electrode(s) during an experiment.

Some of the analysis options transform your data using calculus. Operations such as differentiation or semi-integration change the units on the Y axis. If you use these functions, the signal plotted along the Y axis will no longer represent a current. Instead, the signal will have units of current multiplied by some type of time unit. For example, if you take the derivative of your data, the Y axis units might become \textit{amps per second}. The software automatically keeps up with these changes, and the appropriate units are displayed in the text boxes along the side of the panel.

**The Primary Cursor**

One of the first items you should notice on the graph is the large green cross-hair cursor. (If the cursor is not visible, click on the “CURSOR” button.) This cursor lets you scrutinize individual data points on the graph. You can activate this cursor by clicking on it with the mouse. Once it is active, you can move it around the screen using either the mouse or the arrow keys on the keyboard. No matter where you place the cursor on the graph, it always “snaps” to the nearest data point on the graph.

![Data at Cursor Table]

**FIGURE 24: THE CURSOR DATA DISPLAY**

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Cursor Position

When you move the primary cursor, the text boxes along the side of the panel are updated (see Figure 24). The CURSOR POSITION information lets you know on which data point the cursor is located. The TIME box tells you how long after the start of the experiment the particular data point was acquired. (The start of the experiment corresponds to application of the electrolysis potentials or the start of the sweep program.)

Another box gives you the position of the cursor in terms of the data point number. The point number is expressed two different ways. The line labeled “EXPT” gives the absolute point position within the entire data set. The first data point acquired is point number one, and points are numbered sequentially after that. Note that point number one does not always correspond to time zero.

The other line labeled “TRACE” gives the relative point position with respect to the start of the plot. When the entire data set is plotted on the graph, this line is the same as the one labeled “EXPT.” When a subset of the data is plotted, the two will differ.

The last line of this text box indicates whether or not the graph has been expanded using the “zoom” feature. There are five levels of zooming available, as discussed later, and the zoom level is indicated on this line.

Signal Information

Regardless of which signals appear on the graph or the particular signal the cursor is sitting on, information about all signal levels is posted on the panel. Because the software always maintains two copies of the data set (one ORIGINAL copy and one that can be MODIFIED), there are usually eight signal levels to display. Four text boxes are used to display these signal levels (see Figure 24).

The top two boxes display the MODIFIED data set and the lower two boxes display the ORIGINAL data set. Within each box, the first line is used to indicate the current and the second line indicates the potential. If for some reason, a particular signal level is unavailable, the message “Signal Unavailable” appears on the appropriate line.

The Zooming Cursors

If you want to see your data up close, you can use the zooming features to expand a portion of the graph so that it takes up the entire graph display. Simply click on the ZOOM ON button to activate the zooming cursors. These two dark blue cross-hair cursors run the full width and height of the graph display. Taken together, these two cursors define a rectangular region on the graph that can be expanded to fill the whole graph by clicking on the ZOOM IN button.

You may ZOOM IN up to five times on a particular graph. To go back to the previous display, click on the ZOOM OUT button. To go back to the entire graph
(no zooming) click on the ZOOM OFF button. The zoom level (1 through 5) is indicated in the CURSOR POSITION text box.

Zooming only alters the portion of the graph which appears on the screen. Keep in mind that the active data set still includes portions of the data which may not appear in the zoom region. Analysis functions continue to apply to the entire active data set even if you have zoomed-in on only a portion of the active data.

**Hiding the Cursors**
Occasionally, you may wish to hide the cursors on the graph so that they do not obscure the data. This is usually the case when you are about to print the graph on a hard copy device. Clicking on the CURSOR button allows you to show or hide the cursors.

**Placing a Grid on the Graph**
The GRID button lets you display or hide a grid on the graph. The grid lines are positioned along the axis so that they coincide with the tick marks.

**Clipping Data Using the Primary Cursor**
Occasionally it is desirable to work with only a small portion of the experimental data set. One of the ways to do this is to “clip” out a particular section of the data. To clip a particular range of data, move the primary cursor to the first data point in the range of interest. Then, click on the button labeled CLIP. Next, move the primary cursor to the last data point in the range. Then, click on the same button, which is now labeled CLIP END.

![Image: Clipping Data](image)

**FIGURE 25: CLIPPING A PORTION OF THE DATA SET**
At this point the portion of the data you have selected for clipping is replotted using a heavy stroke (see Figure 25). A dialog box appears which tells you how many points you have selected for clipping. Choose the YES button to go ahead and clip the signal, or press No to leave the signal unaffected.
Once you have clipped a portion of a MODIFIED signal, the rest of that signal is no longer available for analysis or graphing. That is, the clipped portion replaces the signal it was clipped from. Of course, the unaltered ORIGINAL signal remains intact, and you can always retrieve the ORIGINAL signal at any time (see the discussion of the Restore menu in the next section).
Analyzing the Results

The remaining portion of this manual is devoted to a detailed discussion of all of the analysis commands available from the menubar on the ANALYSIS PANEL.

The File Menu

<table>
<thead>
<tr>
<th>File</th>
<th>Plot</th>
<th>Toolbox</th>
<th>Smooth</th>
<th>Restore</th>
<th>Math</th>
<th>Experiment</th>
<th>Instrument</th>
<th>View</th>
</tr>
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<tr>
<td>About PineChem...</td>
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<td></td>
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<tr>
<td>Load Results...</td>
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<td></td>
<td></td>
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<tr>
<td>Save Results...</td>
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<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Import Results...</td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
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<td>Export Results...</td>
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<td>Print Graph</td>
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<td></td>
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</tr>
<tr>
<td>Exit PineChem</td>
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<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
</tbody>
</table>

All commands related to data storage, transfer, and printing are in the File menu. You may store and retrieve files created by this software package using commands found in this menu. You can also export data to other applications or print out a graph of the data.

About PineChem...
This command gives you information about which version of this software package is installed on your system. If your system is presently connected to an AFCBP1 Bipotentiostat, then it gives you the version number of the firmware installed inside the instrument as well.

Load Results...
This command is used to retrieve the results of a previous experiment from a floppy disk or hard drive. A standard dialog box appears which lets you choose which file you wish to load. Files with the suffix “.EXP” are generally used to store experimental results. Any information previously stored in memory is overwritten, but you will be warned before any unsaved data is lost.

Save Results...
This command is used to store information to a floppy disk or hard drive. All information presently in memory is stored in the disk file. This includes both the ORIGINAL and MODIFIED data sets along with all of the parameters defining the experiment. A standard dialog box appears which lets you choose a name for the file being created. Note that the suffix “.EXP” is suggested for files which contain experimental results. If you specify a file name which already exists, a warning message appears before the file is overwritten.
Import Results...
This command is used to import experimental results from files created by an earlier version of this software package. This feature only works with files that were created using the ASWCV1 software package. A standard dialog box appears which lets you choose which file you wish to import. Files created by the ASWCV1 software usually have the suffix “.BIN” as part of the filename. Importing data causes any information previously stored in memory to be overwritten, however, you will be warned before any unsaved data is lost.

No “sweep segment” information is imported from these older “.BIN” files. This means that the Linear Least Squares smoothing option (for smoothing the potential sweep signal) will not work properly with imported data. Also, “pure” setup files created using the ASWCV1 software (i.e., those which do not contain experimental results) cannot be imported.

Export Results...
This command allows you to store data from your experiment in an ASCII format which is readable by most other software packages, including spreadsheet and word processing applications. You may choose which signals are sent to the export file using a dialog box (see Figure 26). A group of check boxes lets you to select only the data that you actually need in the export file. You should make an attempt to limit the amount of information exported so that the export process takes less time.

The general format of the exported file is a series of lines (or rows) that are terminated with a “carriage return & linefeed” or some other “row delimiter” of your choosing. Every data point taken during the experiment occupies a single line in the export file. The information on each line is separated into different fields (or columns) using a “tab” character or some other “column delimiter” of your choosing. There can be quite a number of fields associated with each data point. Typically, these fields include the time at which the data point was acquired, and the currents and potentials at each working electrode at that point in time. You can include the ORIGINAL and/or MODIFIED data in the export file.

Other information can also be included in the export file. If you intend to load the exported file into a spreadsheet or graphing program, it is a good idea to activate the SIGNAL COLUMN TITLES option. This includes a line in the export file which lets you know which data is in which column. This line can be placed at the top or bottom of the data, or it can be omitted entirely.

You may also choose to include an EXPERIMENT RECORD in the export file to describe, in detail, the parameters used to set up the experiment. This record can be included at the top or at the bottom of the export file. Each line in this record represents an individual experimental parameter. There are typically three pieces of information per line: the parameter title, its value, and its units. These three items are separated using the column delimiter (i.e., a “tab” character). Table III shows a typical parameter record for an electrolysis
experiment, while Table IV shows one for a sweep experiment. Please note that the descriptions found in the table are not included in the export file.

**FIGURE 26: EXPORTING EXPERIMENTAL DATA**

Standard scientific notation is used to format the numeric data in the exported file. Time is specified in seconds, potentials are specified in volts, and currents are specified in amperes. If a calculus-based transform has been applied to a current signal, then signal units are of the form amp•sec\(^n\), where \(n\) is some non-zero exponent. The value of \(n\) for both the I1 MODIFIED and I2 MODIFIED signals can be obtained from the EXPERIMENT RECORD if required. It is possible for these two signals to have different values for \(n\). (The signal units for I1 ORIGINAL and I2 ORIGINAL are always amperes.)

**Exporting Data Directly to Microsoft Excel™**

Clicking on the DDE TO EXCEL... button initiates a special data export operation that uses Dynamic Data Exchange™ (DDE) to send data directly to an Excel spreadsheet. This option is only available using the Microsoft Excel 5.0 workbook included with the PineChem software. To make use of this feature, you must have Microsoft Excel 5.0 properly installed and running simultaneously with the PineChem software package. This feature works best on computer systems which have at least an 80486 microprocessor with at least 8 MB of memory. (The DDE feature is slow and unreliable on more minimal systems.)

To establish a link to Excel, you must first load into memory the empty copy of the “PINE.XLS” spreadsheet found in the “PINE” subdirectory on your hard drive. (It must be in this subdirectory). Once you have the “PINE.XLS” spreadsheet loaded, return to the PineChem software package, and click on the DDE TO EXCEL... button. If all goes well, the mouse cursor changes to an
### Table III: Electrolysis Experiment Parameter Record

<table>
<thead>
<tr>
<th>Parameter Title</th>
<th>Parameter Value</th>
<th>Parameter Units</th>
<th>(descriptions)</th>
</tr>
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<tr>
<td>Filename</td>
<td>sample2.exp</td>
<td></td>
<td>original source of exported data</td>
</tr>
<tr>
<td>Experiment Type</td>
<td>Electrolysis</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Data Acquisition</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total Points</td>
<td>6000</td>
<td>points</td>
<td>number of points in ORIGINAL DATA set</td>
</tr>
<tr>
<td>Sampling Interval</td>
<td>+5.00000000E-03</td>
<td>sec</td>
<td>the POINT INTERVAL</td>
</tr>
<tr>
<td>Experiment Length</td>
<td>+3.00000000E+01</td>
<td>sec</td>
<td>the duration of the experiment</td>
</tr>
<tr>
<td>Initial Conditions</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E1 Initial</td>
<td>500</td>
<td>mV</td>
<td>initial potential for K1 electrode</td>
</tr>
<tr>
<td>E2 Initial</td>
<td>0</td>
<td>mV</td>
<td>initial potential for K2 electrode</td>
</tr>
<tr>
<td>Delay Periods</td>
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<tr>
<td>Initial Delay</td>
<td>+3.00000000E+00</td>
<td>sec</td>
<td>duration of induction period</td>
</tr>
<tr>
<td>Final Delay</td>
<td>+1.00000000E+00</td>
<td>sec</td>
<td>duration of relaxation period</td>
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<td>Current Ranges</td>
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<tr>
<td>I1 Maximum</td>
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<td>K1 ELECTRODE SENSITIVITY</td>
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<tr>
<td>I2 Maximum</td>
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<td>K2 ELECTRODE SENSITIVITY</td>
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<td>Other Parameters</td>
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<td></td>
</tr>
<tr>
<td>Run with Dummy?</td>
<td>0</td>
<td>1=Yes; 0=No</td>
<td>Was external cell disengaged?</td>
</tr>
<tr>
<td>Use K2 Electrode?</td>
<td>1</td>
<td>1=Yes; 0=No</td>
<td>Were K2 signals acquired?</td>
</tr>
<tr>
<td>Invert Modified Data?</td>
<td>0</td>
<td>1=Yes; 0=No</td>
<td>Has MODIFIED data set been inverted?</td>
</tr>
<tr>
<td>I1 (mod) sec exponent</td>
<td>+0.00000000E+00</td>
<td>(non-zero)</td>
<td>time unit exponent for I1 MODIFIED</td>
</tr>
<tr>
<td>I2 (mod) sec exponent</td>
<td>+0.00000000E+00</td>
<td>if calculus</td>
<td>time unit exponent for I2 MODIFIED</td>
</tr>
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<td>Electrolysis Potentials</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>E1</td>
<td>-200</td>
<td>mV</td>
<td>K1 electrolysis potential</td>
</tr>
<tr>
<td>E2</td>
<td>0</td>
<td>mV</td>
<td>K2 electrolysis potential</td>
</tr>
</tbody>
</table>

### Table IV: Analog Sweep Experiment Parameter Record

<table>
<thead>
<tr>
<th>Parameter Title</th>
<th>Parameter Value</th>
<th>Parameter Units</th>
<th>(descriptions)</th>
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<tbody>
<tr>
<td>Filename</td>
<td>sample1.exp</td>
<td></td>
<td>original source of exported data</td>
</tr>
<tr>
<td>Experiment Type</td>
<td>Dual Sweep/Analog/Collection</td>
<td></td>
<td>Collection, Shielding, or Window</td>
</tr>
<tr>
<td>Data Acquisition</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Total Points</td>
<td>840</td>
<td>points</td>
<td>number of points in ORIGINAL DATA set</td>
</tr>
<tr>
<td>Sampling Interval</td>
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<td>sec</td>
<td>the POINT INTERVAL</td>
</tr>
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<td>Experiment Length</td>
<td>+8.40000000E+00</td>
<td>sec</td>
<td>the duration of the experiment</td>
</tr>
<tr>
<td>Initial Conditions</td>
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<td></td>
</tr>
<tr>
<td>E1 Initial</td>
<td>500</td>
<td>mV</td>
<td>initial potential for K1 electrode</td>
</tr>
<tr>
<td>E2 Initial</td>
<td>0</td>
<td>mV</td>
<td>initial potential for K2 electrode</td>
</tr>
<tr>
<td>Delay Periods</td>
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<td></td>
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<tr>
<td>Initial Delay</td>
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<td>sec</td>
<td>duration of induction period</td>
</tr>
<tr>
<td>Final Delay</td>
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<td>sec</td>
<td>duration of relaxation period</td>
</tr>
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<td>+5.00000000E-04</td>
<td>A</td>
<td>K1 ELECTRODE SENSITIVITY</td>
</tr>
<tr>
<td>I2 Maximum</td>
<td>+1.00000000E-06</td>
<td>A</td>
<td>K2 ELECTRODE SENSITIVITY</td>
</tr>
<tr>
<td>Other Parameters</td>
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<td></td>
<td></td>
</tr>
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<td>Run with Dummy?</td>
<td>0</td>
<td>1=Yes; 0=No</td>
<td>Was external cell disengaged?</td>
</tr>
<tr>
<td>Use K2 Electrode?</td>
<td>1</td>
<td>1=Yes; 0=No</td>
<td>Were K2 signals acquired?</td>
</tr>
<tr>
<td>Invert Modified Data?</td>
<td>0</td>
<td>1=Yes; 0=No</td>
<td>Has MODIFIED data set been inverted?</td>
</tr>
<tr>
<td>I1 (mod) sec exponent</td>
<td>+0.00000000E+00</td>
<td>(non-zero)</td>
<td>time unit exponent for I1 MODIFIED</td>
</tr>
<tr>
<td>I2 (mod) sec exponent</td>
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<td>if calculus</td>
<td>time unit exponent for I2 MODIFIED</td>
</tr>
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<td>K1 Sweep Program</td>
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<td></td>
</tr>
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<td>Sweep Rate</td>
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<td>mV/s</td>
<td>sweep rate</td>
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<tr>
<td>Initial Direction</td>
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<td>1=Up; 0=Down</td>
<td>initial sweep direction</td>
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<tr>
<td>Sweep Total</td>
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<td>sweeps</td>
<td>number of sweep segments</td>
</tr>
<tr>
<td>Initial Potential</td>
<td>500</td>
<td>mV</td>
<td>initial potential for the sweep program</td>
</tr>
<tr>
<td>Final Potential</td>
<td>500</td>
<td>mV</td>
<td>final potential for the sweep program</td>
</tr>
<tr>
<td>Lower Limit</td>
<td>-200</td>
<td>mV</td>
<td>lower sweep limit potential</td>
</tr>
<tr>
<td>Upper Limit</td>
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<td>mV</td>
<td>upper sweep limit potential</td>
</tr>
<tr>
<td>Sweep K1?</td>
<td>1</td>
<td>1=Yes; 0=No</td>
<td>Was sweep applied to K1?</td>
</tr>
<tr>
<td>Sweep K2?</td>
<td>0</td>
<td>1=Yes; 0=No</td>
<td>Was sweep applied to K2?</td>
</tr>
<tr>
<td>K2 held at</td>
<td>0</td>
<td>mV</td>
<td>potential of “other” electrode or K2 offset</td>
</tr>
</tbody>
</table>
hourglass, and data transfer begins. It is recommended that you do not disturb the computer during the transfer. Note that the transfer may take several minutes depending on the amount of data you are exporting.

Once the DDE transfer is complete, you should save the spreadsheet using a name other than PINE.XLS. This preserves the empty PINE.XLS file for use with future transfers.

**Print Graph**
This command sends a copy of the ANALYSIS PANEL graph to the system printer. Your printer must be set up and configured properly for use as the Microsoft Windows default printer. If you experience difficulties while printing, please make certain you have the most up-to-date version of your printer driver installed on your system. Also note that printing can take up to two or three minutes depending on particular type of printer you are using.

If you wish, you may print a copy of the graph to disk. If you do this, a “.PRN” file is placed on your disk that can later be sent to your printer. To transfer such a file to the printer, you must enter the following command from the MS-DOS command line,

```
C> COPY FILENAME.PRN PRN/B
```

where “FILENAME.PRN” is the name of the file to which you printed the graph. Note that the “C>“ is the prompt normally issued to you when you enter the MS-DOS operating system. You are only supposed to type the text that appears above in **UNDERLINE** print. This command causes the “.PRN” file to be copied, byte for byte, to your printer.

**Exit PineChem**
This command exits the software package. If there is any unsaved work still in memory, you are given a last chance to save it before exiting.
## The Plot Menu

<table>
<thead>
<tr>
<th>File</th>
<th>Plot</th>
<th>Toolbox</th>
<th>Smooth</th>
<th>Restore</th>
<th>Math</th>
<th>Experiment</th>
<th>Instrument</th>
<th>View</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Original Data</td>
<td>![Check Mark]</td>
<td></td>
</tr>
<tr>
<td>![Check Mark]</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Modified Data</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>I1 vs. t</th>
<th>I2 vs. t</th>
<th>I1, I2 vs. t</th>
<th>E1 vs. t</th>
</tr>
</thead>
<tbody>
<tr>
<td>![Check Mark]</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>I1 vs. E1</th>
<th>I2 vs. E1</th>
<th>I1 vs. E1, I2 vs. E2</th>
<th>E2 vs. t</th>
</tr>
</thead>
<tbody>
<tr>
<td>![Check Mark]</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>I1 vs. E2</th>
<th>E1, E2 vs. t</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>![Check Mark]</td>
</tr>
</tbody>
</table>

This menu contains commands which change the way in which your voltammogram is graphed on the ANALYSIS PANEL. Given that a dual working electrode experiment generates two independent signals and that the software maintains two copies of your data in memory (ORIGINAL and MODIFIED), there are a great many ways in which the data can be graphed. The software provides thirteen different kinds of graphs, and on each of these, you may view either the ORIGINAL or MODIFIED data sets, or you can overlay both.

The first two menu items are **Original Data** and **Modified Data**, and when you select one of these items, a check mark is placed next to it in the menu. Whichever item is checked determines which data signals (ORIGINAL and/or MODIFIED) are used to produce the graph on the ANALYSIS PANEL.

![Graph Example](image)

**Figure 27: Overlying Original and Modified Data Sets**
If you check both ORIGINAL and MODIFIED, a graph overlaying both data sets appears on the ANALYSIS PANEL. This is the only overlay option provided by the software package. You can use it to directly compare your raw experimental data with the results of any analysis operation you may have performed. For example, Figure 27 depicts some data from an electrolysis experiment. The digitally smoothed MODIFIED data set is plotted on top of a very noisy ORIGINAL data set.

The next group of items in the Plot menu lets you view the signal from the K1 working electrode as a plot of current versus time (I1 vs. t) or current versus either the K1 or K2 electrode potentials (I1 vs. E1 or I1 vs. E2). When plotting current versus potential, it is recommended that you use the potential (E1 or E2) that was actually swept during the experiment. (Otherwise a somewhat strange plot of current versus a constant potential appears.)

The next group of items in the Plot menu are for viewing only the K2 electrode signal. Again, graphs can be made of current versus time (I2 vs. t) or current versus either the K1 or K2 electrode potentials (I2 vs. E1 or I2 vs. E2). These graphs are only meaningful for experiments which involve two working electrodes.

![current versus potential](image1)

![current versus time](image2)

**FIGURE 28: THE TWO VIEWS OF A VOLTAMMOGRAM**

When performing a dual electrode technique (such as rotated ring-disk voltammetry), it is often desirable to view the signals from both working electrodes on the same graph. The next four choices in the Plot menu are the most common ways to plot two signals on the same graph. You should choose the most appropriate plot according to the type of experiment you have performed (collection, shielding, or window).

Finally, the last three items in the menu let you take a look at the working electrode potentials used during the experiment. Normally, this graph is not very interesting, but it may be useful for experiments where you have supplied an externally generated potential waveform.
The Toolbox Menu

This menu contains commands which activate various on-screen tools that you can use to take quantitative measurements directly from the graph display. **Tools are active only when you are viewing your data as a graph of current versus time.** While you are using a tool, most of the other menus are disabled.

Although voltammograms are normally plotted as current versus potential, a plot of current versus time is actually better suited for tool usage. Such a graph spreads out the voltammogram across the screen even if the experiment involves multiple sweeps (*see Figure 28*). This lets you see the whole experiment at a glance, and easily pick out and move to a particular feature of interest.

There are three tools available (peak height, sigmoid height, and peak area), and all three operate on the same basic principles. When a tool is activated, two or more control points appear on your graph. These control points usually define where a measurement is to be made and the location of a baseline against which the measurement is made.

To move a tool around on the graph, you must use the mouse to move the control points around. Each time a control point is moved, the software redraws the tool in its new location and then recalculates whatever quantity is being measured. The measurement result is displayed in a small floating window. A menubar along the top of this window lets you adjust various parameters that control how the tool makes a measurement.

**The Peak Height Tool**

This tool allows you to make an accurate measurement of the height of any peak shaped features in your voltammogram. It is mainly intended for use with cyclic voltammetry, where peak height measurements must be corrected for a non-Faradaic background current; however, the tool is flexible enough to be used in other situations as well.

This tool has three control points (*see Figure 29*). The large green cross-hair is placed on the graph at the exact position where the height of the peak is measured. You may place this control point anywhere you like, but in most cases you will want to put it directly on top of the peak. Use the mouse to move the cross-hair to the top of the peak, and then use the arrow keys for fine adjustment as needed.
The other two control points determine the baseline against which the peak height measurement is made. These control points appear as two small green crosses located along the current vs. time trace. The portion of the data between the two control points is used to approximate the baseline current. The baseline is extrapolated over to the position of the measurement control point. For a typical cyclic voltammogram, you should position the two baseline control points somewhere along the “flat” portion of the voltammogram immediately preceding the peak.

Each time you move any of the control points, the peak height is recalculated and displayed in a floating window on top of the graph. In addition, the extrapolated baseline and a vertical line representing the peak height are drawn on top of the voltammogram.

![Figure 29: The Peak Height Tool](image)

When you are finished using the peak height tool, simply click on the Cancel button to dismiss the tool. The software redraws the graph of your data and reactivates the menubar on the Analysis Panel.

**Baseline Options**

The floating window has a small menubar that contains only one active menu. This **Baseline** menu contains commands that allow you to alter the way in which the software determines the baseline. The default setting is the **Least Squares** option. The baseline is computed by performing a conventional linear least squares fit to the portion of the current trace that lies between the two baseline control points. This option is probably the best one to use when you have a well defined voltammogram like that shown in Figure 29.

Another baseline setting is the **Free Form** option, which lets you define any arbitrary baseline using the baseline control points. When this setting is active,
the two baseline control points can be placed anywhere on the screen, and the
line connecting them is used as the baseline.

The third baseline setting is the **X-Axis** option, which simply uses the X-Axis as
the baseline. This is essentially the same as not using a baseline at all.

**Subtracting the Baseline**

Once you have established a satisfactory baseline, you may want to
permanently subtract it from the current signal (see Figure 30). This is a good
idea if you plan to perform an integration or semi-integration on the current vs.
time data because it helps eliminate the background charging current.

![Figure 30: A Voltammogram before and after Baseline Subtraction](image)

Simply click on the **SUBTRACT** button to initiate the baseline subtraction. A dialog
box will appear which lets you know which signal is being affected and how
many data points are to be altered. You are asked to confirm these choices
before the software actually performs the subtraction. Note that this operation
only affects the **MODIFIED** signal.

**The Sigmoid Height Tool**

Signals observed during electrochemical experiments frequently exhibit
sigmoidal shaped features. The response from a rotated disk electrode or a
microelectrode is usually sigmoidal, and various calculus operations on
conventional cyclic voltammograms can also produce data with sigmoidal
features. The parameter of interest in each of these cases is the height of the
sigmoid which, depending on the type of experiment, may be called either the
Levich current, the limiting current, or a plateau current.
The sigmoid height tool provides you with an easy way to measure these currents. Figure 31 shows a sigmoidal signal that was produced by semi-integrating the voltammogram depicted in Figure 30.

![Figure 31: The Sigmoid Height Tool](image)

**Figure 31: The Sigmoid Height Tool**

The sigmoid tool has five control points. The first pair of control points are the **baseline control points**. These are used to determine the baseline against which the sigmoid height is measured. Usually, these two controls are placed on a flat portion of the data just before the sigmoid. Then, the data that lies between the two points is fitted to a straight line (using a linear least-squares method), and the resulting straight line is used as the baseline.

The **plateau control points** are very similar to the **baseline control points**. This pair of points defines a line which rests on the plateau of the sigmoid. Usually the **plateau control points** are placed on a flat portion of the current trace immediately after the sigmoid. Then, a linear least-squares method is used to fit a straight line to the data that falls between the two points.

Together, the plateau line and the baseline can be used to evaluate the vertical height of the sigmoid, which is simply the vertical distance between these two lines. For an ideal sigmoid, these two lines are exactly parallel, and the vertical distance between them can be measured at any point along the voltammogram.

In reality, however, the lines are rarely parallel, meaning that the height of the sigmoid depends upon where the measurement is made. The software normally makes this measurement at the **inflection point** near the center of the sigmoid. If you wish, you can manually override this default and make the measurement at any point along the voltammogram.
Each time one of the control points is moved, the height of the sigmoid is recalculated. The result is displayed in a floating window on top of the graph and is graphically represented by a vertical line on the graph itself. The baseline and the plateau line are also drawn directly on the graph.

**Baseline and Plateau Line Options**

The **Baseline** menu lets you change the way in which the baseline and plateau lines are determined. The default setting is the **Least Squares** option. Two separate linear least-squares fits are applied to the current vs. time data that falls between the *baseline control points* and that which falls between the *plateau control points*. Figure 31 shows a baseline and a plateau line that have been computed in this fashion.

The **Free Form** setting lets you position the baseline and plateau line anywhere you like by simply moving the control points around the screen. Lines connecting each pair of control points are drawn directly on the graph so you can see exactly where the baseline and plateau line are located.

The **Force Parallel** setting forces the plateau line to be parallel with the baseline. This option should only be used with signals that exhibit nearly ideal sigmoidal shape. The baseline is determined using a linear least-squares method, as usual, but the plateau line is forced to be parallel with the baseline. You still need to place the plateau control points on the plateau when using this option.

**Changing Where the Sigmoid Height is Measured**

The **Measure** menu lets you change the point at which the vertical height of the sigmoid is measured. The default setting, **At Inflection**, causes the software to look for the inflection point near the middle of the sigmoid. The position of the *measurement control point* is ignored when using this setting. The software computes the height of the sigmoid at the data point nearest to the inflection point. This method works well for most sigmoids.

Another setting allows you to control where the sigmoid height is measured by placing the *measurement control point* **At any point** along the sigmoid. When doing this, you should still probably make the measurement somewhere near the middle of the sigmoid.

One of the most accepted ways of measuring the height of a sigmoid is to make the measurement at the point where the current is exactly half the height of the sigmoid. This point is called the *half wave* point. If you wish to make this kind of measurement, you must manually find the *half wave* point on your own.

To help you in this task, use the **At half wave** option found in the **Measure** menu. This causes the software to draw a third line on the graph which is exactly half way between the baseline and the plateau line. The point where this
third line intersects the middle of the sigmoid is the half wave point. All you have to do is manually place the measurement control point at this intersection, and the software computes the sigmoid height at the half wave.

**The Peak Area Tool**

Voltammograms are usually recorded by monitoring the current at a working electrode as a function of time. If this current vs. time data is integrated, it represents a measure of electrical charge. The peak area tool lets you measure the area under a curve or peak in your voltammogram. This tool is designed for use with cyclic voltammetry and/or stripping voltammetry data, but it is flexible enough to be used in other situations as well.

The peak area tool has four control points. The first pair of control points determines which portion of the signal is integrated. These integration limit controls appear as two green cross-hairs that you may position anywhere along the voltammogram. The second pair of control points determines the baseline against which the integration is performed. The baseline control points operate in a fashion similar to those for the peak height tool, described above.

Each time you move one of the control points, the area under the current trace is recalculated and displayed in a floating window on top of the graph. In addition, the extrapolated baseline and a shaded area representing the peak area are placed on the graph (see Figure 32).

When you are finished using the peak area tool, simply click on the CANCEL button to dismiss the tool. The software redraws the graph of your data and reactivates the menubar on the ANALYSIS PANEL.

**Baseline Options**

The floating window has a small menubar that contains only one active menu. This **Baseline** menu contains commands that allow you to alter the way in which the software determines the baseline. The default setting is the **Least Squares** option. The baseline is computed by performing a conventional linear least squares fit to the portion of the current trace that lies between the two baseline control points. This option is probably the best one to use when you have a well defined voltammogram like that shown in Figure 32.

Another baseline setting is the **Free Form** option, which lets you define any arbitrary baseline using the baseline control points. When this setting is active, the two baseline control points can be placed anywhere on the screen, and the line connecting them is used as the baseline.

The third baseline setting is the **X-Axis** option, which simply uses the X-Axis as the baseline. This is essentially the same as not using a baseline at all.
The area under the curve is expressed in coulombs when it is appropriate. The shaded area under the curve represents the integration result.

Click here to permanently subtract the baseline.

These are the baseline control points.

**Figure 32: The Peak Area Tool**

**Subtracting the Baseline**
As with the Peak Height tool, you may permanently subtract the baseline by clicking on the **SUBTRACT** button.
The Smooth Menu

This menu contains commands which can be used to smooth the signals in the MODIFIED data set. They do not alter the ORIGINAL data set. The menu is divided into two submenus, one for Current signals and the other for Potential signals. Although digital smoothing improves the appearance of your data, it should be used with caution as it always reduces the information content of your data. Over-smoothing can seriously distort the MODIFIED data set.

If you are new to the field of computerized data acquisition, digital smoothing may be an unfamiliar concept. You may be used to recording voltammograms with an analog plotter and observing very nice, smooth signals being traced out on the plotter paper. Most modern analog plotters quietly filter out high frequency noise using a built-in electronic filter. Although all measurements have some inherent noise level, this noise usually goes unobserved when recording voltammograms using an analog plotter.

However, when a computer is used to digitally record a voltammogram, the noise is often readily apparent. This is because no electronic filter is placed between the bipotentiostat and the computer. This means that the raw or ORIGINAL data acquired by the software may appear noisy. When using one of the lower current ranges, the noise may be quite significant. Most often, the noise is caused by power line interference and can be greatly reduced by simply shielding the electrochemical cell.

Another way to reduce noise is to use a software algorithm to digitally smooth the data after it has already been acquired and stored in your computer's memory. A number of these algorithms have been included in this software package, and each one is described in more detail below. Both the potential and the current signals may be smoothed, and different methods are used for each.

You may smooth your data as many times as you like. Each time you perform a smoothing operation, you select the type of smoothing algorithm to be used and the signals you wish to smooth (i.e., I1 MODIFIED, I2 MODIFIED, E1 MODIFIED, and/or E2 MODIFIED). If you inadvertently “over-smooth” a signal, remember that you can use a command in the Restore menu to go back to the ORIGINAL data.
Current ▶ Average
This command activates the DIGITAL SMOOTHING panel (see Figure 33) and permits you to apply an averaging algorithm to either or both of the current signals (I1 MODIFIED or I2 MODIFIED). This smoothing algorithm is perhaps one of the easier smoothing methods to understand. Each data point is smoothed by averaging its value with those of neighboring data points.

You can choose the number of data points involved in the average. An equal number of neighboring data points from either side of the data point being smoothed are used to compute the average. Thus, the total number of points averaged is always an odd number greater than two. You choose the number of points using the SMOOTHING INTERVAL control. The greater the number of points, the more your data is smoothed.

You may apply the smooth to one or both current signals. Two check boxes on the DIGITAL SMOOTHING panel are used to select which signals are smoothed. The signals appearing on the ANALYSIS PANEL graph are selected by default.

The averaging method is simple and straightforward but has two disadvantages. First, very sharp features are smoothed away. Second, the very beginning and very end of the data set cannot be smoothed due to insufficient neighboring data. Thus, each time you apply this type of smooth to your data, you lose data points off of each end of the signal.

Current ▶ Savitsky-Golay
This command activates the DIGITAL SMOOTHING panel (see Figure 33) and permits you to apply a well-known Savitsky-Golay smoothing algorithm to either or both of the current signals (I1 MODIFIED or I2 MODIFIED). This smoothing algorithm works by fitting small portions of your data to a local cubic polynomial function.
You can adjust the number of data points involved in the polynomial fit. An equal number of neighboring points are taken from either side of the data point being smoothed. Thus, the total number of points involved is always an odd number greater than two. You choose the number of points using the SMOOTHING INTERVAL control. The greater the number of points, the more your data is smoothed. This method causes some attenuation of peak currents. It is based on the work of A. Savitzky and M. J. E. Golay. You may consult _Analytical Chemistry_, volume 36, page 1627 (1964) for more details.

**Current ► Fourier**

The Fourier method is very powerful because it allows you to remove noise in a surgical fashion. The current vs. time signal is transformed from the time domain to the frequency domain. In the frequency domain, the useful part of the current signal is usually found at lower frequencies while the noisy part of the current signal is at higher frequencies. By viewing your data in the frequency domain, it is an easy matter to eliminate the higher frequency portion of the signal (i.e., the noise) while retaining the lower frequency component of the signal. Once the noise is removed, the signal is then transformed back to the time domain, and the result is a smoothed current vs. time signal. The results can be rather dramatic as illustrated in Figure 34.

![Before and after smoothing](image)

**FIGURE 34: A FOURIER TRANSFORM SMOOTHING EXAMPLE**

The particular implementation of the Fourier smoothing method used by this software package lets you specify a range of frequencies to be removed from the signal. This is sometimes called a Fourier “notch filter” because you are removing a narrow band of frequencies that can be thought of as a notch. Most often, an electrochemical experiment is subject to power line noise at or near a multiple of 60 Hz. (In some countries the power line noise is at multiples of 50 Hz.) Simply removing the frequency components of the current signal that lie between 55 Hz and 65 Hz often produces a satisfactory smooth.

When you choose the **Fourier** command from the **Smooth** menu, a special FOURIER SMOOTHING panel appears on the screen (see Figure 35). Most of the space on this panel is occupied by a large graph. The graph depicts a frequency domain plot of the current signal. The plot is produced by transforming a single segment of the current vs. time data to the frequency domain. The size of the segment is indicated in the lower left-hand corner of the
display. (The number of points in a segment is determined by the sweep program used to perform the experiment and may vary from one segment to the next.)

The X-axis on the graph spans the frequency range from 0.0 Hz up to the so-called Nyquist frequency for your data set. The Nyquist frequency is simply half the sampling frequency (i.e., half of the reciprocal of the POINT INTERVAL). The useful portion of the current signal typically has a large amplitude and appears on the left-hand side of the graph (at lower frequencies). The noise may or may not be visible at higher frequencies.

In the example depicted in Figure 35, it is safe to say that the useful portion of the signal lies at frequencies below 25 Hz. Everything above 25 Hz is probably just noise, and the feature near 60 Hz is almost definitely power line noise. A conservative notch filter spanning the range from 55 Hz to 65 Hz (as shown) would remove the 60 Hz feature and should greatly improve the appearance of the current signal. A less conservative approach might be to remove everything from 25 Hz up to the Nyquist frequency. You may find that it is best to engage in a bit of trial-and-error to find the frequency range that works best for your data.

Two vertical red lines on the graph are cursors which specify the range of frequencies in the notch filter. You should move these cursors so that they bracket the range of frequencies you wish to remove from your current signal.

**FIGURE 35: THE FOURIER SMOOTHING PANEL**

In the example depicted in Figure 35, it is safe to say that the useful portion of the signal lies at frequencies below 25 Hz. Everything above 25 Hz is probably just noise, and the feature near 60 Hz is almost definitely power line noise. A conservative notch filter spanning the range from 55 Hz to 65 Hz (as shown) would remove the 60 Hz feature and should greatly improve the appearance of the current signal. A less conservative approach might be to remove everything from 25 Hz up to the Nyquist frequency. You may find that it is best to engage in a bit of trial-and-error to find the frequency range that works best for your data.

Two vertical red lines on the graph are cursors which specify the range of frequencies in the notch filter. You should move these cursors so that they bracket the range of frequencies you wish to remove from your current signal.
The specific location of these cursors along the frequency axis is indicated in the upper right-hand corner of the panel.

The Fourier method usually does not attenuate peak heights, but it can introduce artificial oscillations known as “ringing” into the current vs. time signals. Ringing is usually an indication that you have used a notch filter which has removed some of the useful portion of the signal. In this case, you should move the notch filter so it starts at a higher frequency.

Clicking on the SUGGEST button causes the software to choose an appropriate range of frequencies to remove. The method it uses to determine this range works well with most data sets, but the final decision still lies with you. Note that the software always suggests a notch which is bounded on the high end by the Nyquist frequency.

Viewing Different Segments
Rather than smoothing the entire current signal at once, the Fourier smooth applies the notch filter repeatedly to smaller segments of your data. These smaller segments correspond exactly with the sweep segments from the potential sweep program. The graph on the FOURIER SMOOTHING panel depicts the transform of just one segment at a time. When you are deciding which frequencies to use for your notch filter, it may be useful to take a look at the transforms of several different segments. Two controls on the panel allow you to specify which portion of which signal appears on the graph.

The first of these GRAPH APPEARANCE controls lets you change the SIGNAL being viewed. You may view transforms of any of these four data sets: I1 MODIFIED, I2 MODIFIED, I1 ORIGINAL, and I2 ORIGINAL. (Of course, you can only smooth the MODIFIED data sets.)

The second GRAPH APPEARANCE control lets you specify which SEGMENT of the signal is displayed on the graph. Usually segments near the middle of the sweep program are more representative of the overall data set. (If you have clipped your data, some segments may be unavailable for viewing.)

Performing the Smooth
Once you have determined the frequencies for your notch filter, you can apply the filter to the I1 MODIFIED signal and/or the I2 MODIFIED signal. (You may not smooth any of the ORIGINAL signals.) Two check boxes in the lower right-hand corner of the FOURIER SMOOTHING panel let you smooth either or both of these MODIFIED current signals.

To initiate the smoothing process, click on the SMOOTH button. You can watch as each segment of your data is plotted on the graph and then smoothed. During this process you will probably notice that the number of data points in
each segment varies. The notch frequencies that you have chosen may also vary slightly to match the number of points in a particular segment.

If you decide not to perform the smooth, you can click on the CANCEL button. The current signals will not be altered.

**Potential ► Average**

This command activates the DIGITAL SMOOTHING panel (see Figure 33) and permits you to apply an averaging algorithm to either or both of the potential signals (E1 MODIFIED or E2 MODIFIED). This algorithm has already been described above in the context of smoothing current signals. When applying it to potential signals, you should be aware that the sharp features found in a potential sweep program may be significantly “rounded-off” (see Figure 36).

![Figure 36: Smoothing Sharp Features](image)

**Potential ► Least Squares**

This smoothing method is geared more toward sweep experiments. It takes advantage of the way in which the sweep program can be divided into a set of linear sweep segments. The data from each sweep segment is fitted using a linear least squares analysis, and the best straight line describing the segment is used to generate smoothed data (see Figure 37). The very sharp features between sweep segments are retained, and all of the noise is effectively eliminated.

![Figure 37: Least Squares Smoothing of the Potential Signal](image)

**Special Note:** This smoothing method does not work properly with imported data files created using version 1.01 of this software package.
The Restore Menu

This menu contains commands for copying signals from the ORIGINAL data set into the MODIFIED data set. You can use these commands whenever you have hopelessly corrupted all or part of the MODIFIED data set (by oversmoothing, accidentally multiplying it by zero, etc.). These commands are explained in more detail below.

**All Signals**
This command replaces the contents of the entire MODIFIED data set with the ORIGINAL data acquired during the experiment. This essentially restores the file to the point just after the experiment concluded and before any analysis was performed.

**Entire I1 Signal**
This command copies the entire I1 ORIGINAL signal into the I1 MODIFIED signal. Any previous data clipping or analysis performed on the I1 MODIFIED signal is lost. No other MODIFIED signal is affected.

**Entire I2 Signal**
This command copies the entire I2 ORIGINAL signal into the I2 MODIFIED signal. Any previous data clipping or analysis performed on the I2 MODIFIED signal is lost. No other MODIFIED signal is affected.

**Entire E1 Signal**
This command copies the entire E1 ORIGINAL signal into the E1 MODIFIED signal. Any previous data clipping or analysis performed on the E1 MODIFIED signal is lost. No other MODIFIED signal is affected.

**Entire E2 Signal**
This command copies the entire E2 ORIGINAL signal into the E2 MODIFIED signal. Any previous data clipping or analysis performed on the E2 MODIFIED signal is lost. No other MODIFIED signal is affected.

**Clipped I1 Signal**
This command restores only the clipped portion of the I1 MODIFIED signal using the corresponding data points from the I1 ORIGINAL signal. Any analysis performed on the I1 MODIFIED signal is lost, but the data clip boundaries remain
unchanged. If the signal has not been clipped, then this command has the same effect as the **Entire I1 Signal** command described above. No other **MODIFIED** signal is affected.

**Clipped I2 Signal**
This command restores only the clipped portion of the I2 **MODIFIED** signal using the corresponding data points from the I2 **ORIGINAL** signal. Any analysis performed on the I2 **MODIFIED** signal is lost, but the data clip boundaries remain unchanged. If the signal has not been clipped, then this command has the same effect as the **Entire I2 Signal** command described above. No other MODIFIED signal is affected.

**Clipped E1 Signal**
This command restores only the clipped portion of the E1 **MODIFIED** signal using the corresponding data points from the E1 **ORIGINAL** signal. Any analysis performed on the E1 **MODIFIED** signal is lost, but the data clip boundaries remain unchanged. If the signal has not been clipped, then this command has the same effect as the **Entire E1 Signal** command described above. No other MODIFIED signal is affected.

**Clipped E2 Signal**
This command restores only the clipped portion of the E2 **MODIFIED** signal using the corresponding data points from the E2 **ORIGINAL** signal. Any analysis performed on the E2 **MODIFIED** signal is lost, but the data clip boundaries remain unchanged. If the signal has not been clipped, then this command has the same effect as the **Entire E2 Signal** command described above. No other MODIFIED signal is affected.

**The Math Menu**

The first four commands in this menu let you perform various mathematical operations on the I1 **MODIFIED** and/or I2 **MODIFIED** signals. These commands do not alter the **ORIGINAL** data set. All four of these commands cause the standard **MATH FUNCTION** panel to appear on the screen (see Figure 38). This panel lets you choose which signal(s) are to be affected by the math operation and reminds you how many data points comprise each of these signals. To initiate the math operation, click on the OK button. Click on the CANCEL button to leave your data unchanged.
Scale and Offset...
This command allows you to scale (multiply) either or both current signals by a constant. You can also use it to add an offset constant to either or both signals. Both the scale and offset operations may be performed simultaneously.

When you first select this command, the standard Math Function panel appears, and you can use the controls on this panel to determine which signal(s) to alter. After you click on the OK button, a second panel appears. Enter the scaling factor and offset value using this panel and then click on the OK button.

Semi-Integrate...
This command semi-integrates either or both current signals with respect to the square root of time. The current response observed in most voltammetric experiments is governed by the rate of physical diffusion of a chemical species in solution, and the rate of diffusion is fundamentally related to the square root of time. The semi-integration operation is often used to transform an electrochemical signal so that this underlying time dependence is removed from the signal. A series of journal articles by K. Oldham describes the advantages of this approach. (see, for example, Analytical Chemistry, vol 44, 1972, page 196.)

If a traditional cyclic voltammogram is subjected to semi-integration, its shape is transformed to a sigmoid. This sigmoidal signal is similar to that produced by classic polarography or a rotated disk electrode experiment.

Semi-integration is a time consuming process that is not recommended for large data sets. You should clip the relevant portion of your data before semi-integrating it. You may also wish to perform a baseline subtraction prior to semi-integration.

Note that the signal units are altered when you perform a semi-integration. Semi-integration of current data results in a new data set with units of amp $\cdot$ sec$^{1/2}$. The new units are reflected in the DATA AT CURSOR section of the
ANALYSIS PANEL. Also note that semi-integration of the same data set twice in a row is the same as performing a single (complete) integration.

**Derivative...**
This command takes the first derivative of either or both current signals with respect to time. Note that the current signal units are altered when you take the derivative. Taking the first derivative of current data results in a new data set with units of amp/sec. These new units are reflected in the DATA AT CURSOR section of the ANALYSIS PANEL.

**Integrate...**
This command integrates the entire data set with respect to time. Note that the current signal units are altered when you integrate the signal. Integration of current data results in a new data set with units of amp•sec (or coulombs). These new units are reflected in the DATA AT CURSOR section of the ANALYSIS PANEL. The primary reason for integrating a voltammogram is to obtain a measure of the amount of electrical charge passed during the experiment.

**Invert Signals**
This command takes the entire MODIFIED data set (currents and potentials) and multiplies it by negative one (−1). This has the effect of flipping the graph both horizontally and vertically. The main reason for performing this operation is to view a voltammogram in the “American” fashion rather than the “European” fashion (which is the default).

If you do invert your data set, be absolutely sure to remember that the labels on the axes are not inverted! This means that a peak which is really at +300 mV will appear on the graph at −300 mV once the data is inverted. A check mark will appear next to the Invert Axes menu item to help remind you that your data is inverted. The ORIGINAL data is not inverted by this command.

**The Experiment Menu**

<table>
<thead>
<tr>
<th>File</th>
<th>Plot</th>
<th>Toolbox</th>
<th>Smooth</th>
<th>Restore</th>
<th>Math</th>
<th>Experiment</th>
<th>Instrument</th>
<th>View</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Electrolysis...</td>
<td>Analog Sweep Voltammetry...</td>
<td>Repeat...</td>
</tr>
</tbody>
</table>

The commands in this menu are used to initiate a new experiment. This menu is always present on every menubar. As of this software release, there are two types of experiments that can be performed.

The **Analog Sweep Voltammetry** experiment can be used to perform a wide variety of electrochemical techniques, such as cyclic voltammetry, anodic stripping voltammetry, and various hydrodynamic methods such as rotated disk voltammetry. The **Electrolysis** experiment is used with experiments where electrode potentials are held constant or those in which an external potential waveform is supplied directly to the Bipotentiostat.
The Repeat experiment command is a special command that appears only in the menubar on the ANALYSIS PANEL. This command lets you perform a new experiment using the exact same parameters which produced the data you are presently analyzing.

To begin a new experiment, the software must clear out any data that is still in memory from a prior experiment. If the data from the prior experiment has not yet been saved to disk, then the message box shown below appears on the screen. This box appears whenever unsaved results are in danger of being overwritten.

![Figure 39: Warning Box for Unsaved Data](image)

You have the option of saving the data to your hard drive, discarding the data, or canceling the operation. If you choose CANCEL, then the data from the previous experiment remains in memory.

The Instrument Menu

The Instrument menu is useful only when you are operating this software package on a computer system that is physically connected to a Bipotentiostat. The Instrument menu appears on every menubar and contains commands which let you monitor the Bipotentiostat and its communications with your computer.

**Status...**
This command activates the INSTRUMENT STATUS panel. Typing “control-l” on the keyboard is a short-cut to this menu command. The INSTRUMENT STATUS panel is described in more detail earlier in the manual. It allows you to control the idle conditions that are applied to the electrochemical cell in between experiments.

**Device List...**
This command displays the SYSTEM HARDWARE DEVICES panel which indicates which Interface Boards and instruments connected to your computer system are recognized by this software package. This panel is mainly useful when you have more than one Interface Board installed in your system. In this case, you must
use the controls on the panel to select which Interface Board you want to use. The software then attempts to connect to a Bipotentiostat attached to the board.

If your system has only one Interface Board (and only one Bipotentiostat), and if you have configured your Interface Board properly as described in the Installation Manual, then as the software starts, it automatically connects to the Bipotentiostat. Displaying the SYSTEM HARDWARE DEVICES panel is not necessary unless you just wish to confirm the Interface Board configuration.

**Reconnect**

If, for any reason, communication between the computer and the Bipotentiostat is interrupted, this command can be used to reestablish contact with the instrument. You are notified whether or not the attempt to reconnect is successful.

**The View Menu**

<table>
<thead>
<tr>
<th>File</th>
<th>Plot</th>
<th>Toolbox</th>
<th>Smooth</th>
<th>Restore</th>
<th>Math</th>
<th>Experiment</th>
<th>Instrument</th>
<th>View</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Potential Program...</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Experiment Parameters...</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Potential Program...**

This command displays the POTENTIAL PROGRAM panel. A list on this panel shows how the data from your experiment is broken up into various segments according to the potential program applied to the working electrode(s) during the experiment (see Figure 40).

For sweep experiments, the list is a kind of directory that tells you exactly which data points fall within a particular sweep segment. It also provides you with an accurate measure of the sweep rate for each segment of the sweep program.

For electrolysis experiments, there are no more than two data segments. The first segment (if present) consists of any data points that were acquired before the onset of electrolysis. The remaining data from the experiment comprises the second data segment.

The example in Figure 40 is taken from a collection experiment. During this experiment, the K1 working electrode potential was swept back and forth between +500 mV and –200 mV a total of six times. The sweep rate was about 485 mV/sec. The K2 working electrode potential was held constant at zero volts.

The DATA RANGE column lists the sweep segment number and the range of data points that fall within the sweep segment. Each sweep segment in the example experiment contains about 286 data points.

The column of “check marks” to the left of the DATA RANGE column indicates which sweep segments are presently being displayed on the ANALYSIS PANEL graph. When working with an experiment that has many segments, you can
reduce the “clutter” on the ANALYSIS PANEL graph by displaying only a few pertinent data segments. Simply check (or uncheck) the appropriate segments and click on the OK button.

The ANALYSIS PANEL graph is redrawn using only those segments you have selected. Thus, your choices act as a kind of filter to determine which portion of the data appears on the graph. Keep in mind, however, that most analysis operations (such as math or smoothing functions) continue to apply to the entire signal (all segments) even though only a subset of the data now appears on the graph.

Perhaps the most useful information in the directory is the actual value for the sweep rate used in each individual sweep segment. These values are listed in the K1 POTENTIAL PROGRAM column and/or the K2 POTENTIAL PROGRAM column. This sweep rates are obtained by performing a linear least squares fit to the potential vs. time data from your experiment. The sign on the sweep rate indicates whether the sweep was anodic (+) or cathodic (–). Note that there is sometimes a small discrepancy between the anodic sweep rate and the cathodic sweep rate. This may cause the number of data points in anodic and cathodic sweep segments to differ by one or two points.

![Potential Program Panel](image)

**FIGURE 40: THE POTENTIAL PROGRAM PANEL**

**Special Note:** Imported data files created by version 1.01 of this software package will not provide a proper potential program listing.

**Experiment Parameters...**

This command displays a list of selected experimental parameters associated with the data set presently in memory. Consult Tables III and IV for more information regarding the meaning of these parameters.
Software Specifications

Table V lists the specifications for the ASWCV2 PineChem Software Package. These specifications are based on version 2.00 of this software and Interface Board specifications as of the date of this manual. All specifications are subject to change in future software releases and as newer technologies become available.

Table V: ASWCV2 PineChem Software Package Specifications

<table>
<thead>
<tr>
<th>Item</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>GENERAL</strong></td>
<td></td>
</tr>
<tr>
<td>Potential Input Ranges:</td>
<td>±10.0 V, ±5.0 V, and ±2.5 V</td>
</tr>
<tr>
<td>Current Input Ranges:</td>
<td>22 ranges from ±1.0 A down to ±100 nA</td>
</tr>
<tr>
<td>A-to-D Conversion:</td>
<td>12 bit (1 part in 4096)</td>
</tr>
<tr>
<td>Minimum Point Interval:</td>
<td>42 µs (4 channel) or 22 µs (2 channel)</td>
</tr>
<tr>
<td>Maximum Point Interval:</td>
<td>655.35 sec (10.92 min)</td>
</tr>
<tr>
<td>Maximum Number of Points:</td>
<td>200000*</td>
</tr>
<tr>
<td><strong>ELECTROLYSIS PARAMETERS</strong></td>
<td></td>
</tr>
<tr>
<td>Working Electrodes:</td>
<td>1 or 2</td>
</tr>
<tr>
<td>Initial and Electrolysis Potentials:</td>
<td>±10 V (in 4.88 mV increments)</td>
</tr>
<tr>
<td>Induction Period:</td>
<td>0 to 300 sec</td>
</tr>
<tr>
<td>Electrolysis Period:</td>
<td>200 µs to 1 year*</td>
</tr>
<tr>
<td><strong>ANALOG SWEEP VOLTAMMETRY PARAMETERS</strong></td>
<td></td>
</tr>
<tr>
<td>Working Electrodes:</td>
<td>2 (either or both may be swept)</td>
</tr>
<tr>
<td>Sweep Rate:</td>
<td>5 to 9995 mV/s (in 5 mV/s increments)</td>
</tr>
<tr>
<td>Number of Sweep Segments:</td>
<td>1 to 60000</td>
</tr>
<tr>
<td>Initial, Limit, and Final Potentials:</td>
<td>±10 V (in 4.88 mV increments)</td>
</tr>
<tr>
<td><strong>ANALYSIS TOOLS</strong></td>
<td></td>
</tr>
<tr>
<td>Plot Options:</td>
<td>I1 and/or I2 versus E1, E2 or time;</td>
</tr>
<tr>
<td></td>
<td>E1 and/or E2 versus time</td>
</tr>
<tr>
<td>Measuring Tools:</td>
<td>Peak Height and Area, Sigmoid Height</td>
</tr>
<tr>
<td>Smoothing:</td>
<td>Least Squares, Fourier, Savitsky-Golay, Averaging</td>
</tr>
<tr>
<td>Math Functions:</td>
<td>Scale, Offset, Derivative, Integration,</td>
</tr>
<tr>
<td></td>
<td>Semi-integration</td>
</tr>
<tr>
<td>Data Export:</td>
<td>Generic ASCII, DDE to Microsoft Excel ™</td>
</tr>
</tbody>
</table>

* These upper limit specifications are estimates based on systems with at least 12 MB RAM. Actual upper limits may vary and depend strongly on the system's memory configuration. Also, systems purchased prior to November 1995 were shipped with the AT-MIO-16H-9 INTERFACE BOARD, which is limited to 16380 points.