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Model 270/250

Research Electrochemistry Software

User's Guide

222153-A-MNL-B

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1 Chapter One Introduction

Overview

The EG&G Princeton Applied Research Model 270/250 Research Electrochemistry Software is designed to perform both qualitative and quantitative electroanalytical experiments quickly, easily, and reliably, using a variety of techniques from cyclic voltammetry to controlled potential coulometry. After your experiments, you can easily analyze your data to determine integrated area, peak height, and full-width at half height. Likewise, it is possible to subtract or just ignore background currents from your data set.

Extensive graphics capabilities are built into the software, including real-time plotting during experiments, plot overlay for data comparison, and digital graphics printing. Real-time plotting lets you monitor the measurement progress, and easy-to-use plotting routines let you rescale and overlay curves on the screen. You can then produce multicolor hard copies on a digital plotter with just a click of the mouse. One particularly useful feature is the straightforward, easy-to-use plot editing.

All these capabilities are available using an IBM compatible personal computer and a GPIB card. The host computer provides memory, data processing, input-output, and interfacing between the operator and the system.

Note that in the past the Model 270 and Model 250 were separate programs that controlled different potentiostats. These packages have been combined into the Model 270/250 software. For simplicity, this manual will generally refer to the Model 270/250 software as just the Model 270 or 270.

The Model 270 software can control the EG&G PARC VersaStat (Model 253) and the Models 263, 273, 273A, and 173/276 potentiostat/galvanostats. It can use each of these instruments to its fullest capability. A printer or plotter, or both, and an electrochemical cell complete the system. In addition, the software can also use the 273 or 263 to control the EG&G PARC Model 303A Static Mercury Drop Electrode or use the Model 273 to control the Model 616 Rotating Disk Electrode.

Convenience

The software is designed to be as user-friendly as we could make it. Banner and pulldown menus give you easy access to all system functions. To select a pulldown menu or a measurement parameter, all you have to do is click your mouse on the selection, or enter the first letter of the selection from the keyboard.

The Model 270 Software automates all of the subtasks involved in running an experiment. Under control of your menu selections, it automatically controls the potentiostat, acquires the data, and plots it on the display screen. Default parameter values often allow you to perform a productive experiment with minimal entry of setup values.

Data from an experiment can be stored and is automatically plotted on the screen. Stored data can be recalled and viewed at any time. You can select formatting options such as automatic or manual scaling, connected or discrete data point display, and automated or user-generated axis labels.

Help

You can call up context-sensitive help messages by simply pressing the **<F1>** key or selecting Help on a banner. Without opening a book, you get a brief introduction

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to the function you have selected or the input prompt you are responding to.

Measurement Techniques

The versatile design of the Model 270 software allows you to apply any of the following different experimental techniques:

- Voltammetry
  - Linear Sweep
  - Cyclic Voltammetry
  - Square Wave

- Voltammetry/Polarography
  - Differential Pulse
  - Normal Pulse
  - Reverse Normal Pulse

- Time Based
  - Controlled Potential Coulometry
  - Chronoamperometry
  - Chronocoulometry (requires 273 with /96 option or 273A)
  - Chronopotentiometry

These techniques are discussed in Chapters 14-23. Chapter 4 includes an overview of software operation, and concludes with a sample Linear Sweep Voltammogram experiment designed to check out your setup and serve as a tutorial to get you going.

Data Analysis

The Model 270 software can perform three types of calculations:

- Integration to determine the area under the curve.

- Peak location & calculation of the full-width at half-peak maximum.

- Linear regression analysis.

These techniques are explained in Chapter 7.

The user can easily control the limits of these analyses, although the software may default the analysis to the limits you desire.

In addition, if you've purchased the full Model 270 package, as opposed to the Model 250 package, you can also use your CV or SWV files in the Model 271 Cool Kinetic Analysis Software to determine rate constants associated with nine different mechanistic models. See your Model 271 manual or your EG&G Salesperson for more information.

In addition, the Cut/Paste feature (Chapter 27) allows you to save selected values in text files for use with other programs, such as your word processor or spreadsheet.

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2 Chapter Two System Requirements

Overview

The Model 270 Research Electrochemistry Software uses the Computer Graphics Interface (CGI) from Graphic Software Systems (GSS) to drive peripheral devices. The CGI supports a great variety of commonly used peripherals, including display/adapters, printers, plotters, and mice.

The device drivers you receive with the Model 270 software are a selection of some of the more commonly used GSS drivers. During the software installation, the Install program lists the devices associated with these drivers so that you can select the device you are using.

If you want to use a device not listed by the Install program and you know something about computer peripherals, you can first try selecting a device similar to the one you have. If it doesn't work properly, you may need an additional driver. Contact EG&G PARC to see if the driver you need is available.

The hardware requirements listed in this chapter are meant to be general. For example, the section on plotters states that any plotter using the HPGL graphics language will work. Use this chapter to determine whether your hardware will work with the GSS drivers that we supply.

Computer

Required:

- Any IBM compatible computer (8088 up to 80486 based)
- Hard disk - Minimum of 2 Mbytes free.
- One or more floppy diskette drives (3½" or 5¼")
- 640K conventional plus 512 K expanded or extended RAM
  (the program will run without expanded or extended memory or with less conventional memory, but execution speed may be unacceptable for many applications)

Highly Recommended:

- Math coprocessor
- 1 MB expanded/extended RAM

Computer Operating System

Required: MS-DOS 3.1 or later

Recommended: MS-DOS 5.0

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Monitor and Video Adapter

Required (one of the following):

- MCGA display and adapter
- VGA display and adapter
- EGA display and adapter
- CGA display and adapter
- Monochrome TTL display with Hercules Monochrome Graphics card or compatible

Mouse

Recommended (one of the following):

- Microsoft bus or serial mouse, or compatible
- Logitech bus or serial mouse
- IBM PS/2 mouse or compatible
- PC Systems mouse or compatible

Printer

Recommended (one of the following):

- Dot matrix printer with parallel interface. Supported printers include Epson, IBM, HP DeskJet, HP Paintjet, and Toshiba.
- HP LaserJet II or compatible laser printer with parallel interface
- Postscript printer with serial or parallel interface

A serial printer may be substituted if the DOS MODE command is used to redirect the parallel printer output to a COM port.

Plotter

Required:

- An RS-232 serial interface on plotter. Plotter should be set up for 9600 baud, 8 data bits, 1 stop bit, no parity, and hardware handshaking.

Recommended (one of the following):

- Hewlett-Packard or compatible plotter using the HPGL graphics language
- Houston Instruments or compatible plotter using the DMPL graphics language
- EG&G PARC RE0149 plotter

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GPIB Interface Card

Required (one of the following):

- National Instruments MC-GPIB board
  for IBM PS/2 Microchannel bus computers and compatibles
  Note: The Install program assumes this card is using Interrupt 11. This is ***not*** the default setting. See Appendix C for more information.
- National Instruments PC-2A, PC-2, or PC-AT board
  for IBM PC and compatibles using ISA adapters.

Potentiostat

Required (one or more of the following):

- EG&G PARC Model 273 (firmware version 19 or higher ROMs)
- EG&G PARC Model 273 & /96 Option Board (version 106 or higher ROMs)
- EG&G PARC Model 273A (version 202 or higher ROMs)
- EG&G PARC Model 173 with Model 276 Interface Module
- EG&G PARC VersaStat (Model 253)
- EG&G PARC Model 263

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Overview

The Model 270 software is supplied on both 5¼" and 3½" IBM-formatted floppy disks. You should regard each disk supplied as a master (or archive) disk and store it in a safe place at room temperature, away from dust, moisture, chemicals, and strong magnetic fields. You may duplicate the disks *as required for your own use only.*

Before you install the Model 270 software, set up your hardware as described below. Then, follow the instructions in the Software Installation section on the next page.

Hardware Setup

If you're using a computer or peripherals not purchased from EG&G PARC, check it against the listings in Chapter 2, System Requirements, for compatibility.

First set up your computer. Next install your GPIB board in your computer, leaving all switches set to their default settings. Then cable the GPIB board to your potentiostat. After that, connect the cell cable and power cord to your potentiostat.

Appendix C describes the GPIB address switch settings required for proper communication between your potentiostat and computer. They are usually left at the factory defaults.

Potentiostat Switch Settings

Before turning the system on and starting the program, make sure the potentiostat switches are set as follows:

Model 263:
Cell Type (on Function Menu): DUMMY

VersaStat (Model 253):
Cell Switch: OFF
Galvanostat/Potentiostat Switch: POTENTIOSTAT

Models 273 and 273A:
Cell Enable: OFF
Electrometer Cell Switch: DUMMY

Model 173/276:
Cell selector switch: OFF
Operating Mode: CONTR. E
Meter: Select desired PARAMETER/SCALE
Channel: A

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All other switches: OFF
IR Compensation: OFF
Cell Override: ON (PUSH IN)
Current Range: any

Software Installation

Appendix D lists the files contained on the Model 270 distribution disks. An installation program ("Install") is provided to simplify installation of the Model 270 software. Note that the Install program needs to modify your CONFIG.SYS file to install the appropriate GPIB board driver. The Install program gives you the option of creating a CONFIG.NEW file instead, but in that case you will have to modify your CONFIG.SYS file yourself to make it consistent with the changes in the CONFIG.NEW file.

When you're satisfied that the hardware is properly connected and there is enough room in memory to run properly, simply insert the Install Program Drivers Diskette #1 into drive A: and type:

A: <Enter>

(If you are installing from your B: drive, simply type B: instead. Note that some keyboards have a Return key instead of an Enter key. Both keys work the same way so, if yours says Return, press it whenever this manual says to press Enter.) Now type:

INSTALL <Enter>

You will be prompted for certain information needed by the Install program. Note that you may abort the installation procedure at any time by pressing the <Esc> key.

The Installation program will create an ECHEM.BAT batch file in the root directory of the hard disk. You should use this batch file or one modelled after it to run the software to ensure that the software gets loaded properly into your computer's memory.

You can begin operations as soon as the software is installed. But remember to reboot your computer so DOS can read and execute your newly created CONFIG.SYS file.

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4 Chapter Four Using the Model 270 Software

Overview

When you have completed the Model 270 installation, you're ready to begin operations. This chapter shows you how to start the program and check out your system to ensure that the individual units are communicating.

Before exploring the features and use of the software, however, it's a good idea to review some general operating principles. This chapter provides an introduction to menu navigation, operation using the mouse or the keyboard, the Main Menu selections, how to enter values, and the Help system. It concludes with a sample session using the software to run a Tafel experiment. Detailed operations information starts in Part 2.

Starting the Program

Before applying power to the system, you should carefully review your hardware installation and the switch settings described in Chapter 3. When you are satisfied that all connections and switch settings are correct, use the following procedure to power up.

1. Apply power to all equipment except the potentiostat. That is, turn the power switches on the computer, video monitor, printer, and plotter (if you are using one) to ON.

2. Apply power to the potentiostat.

3. On your computer, enter **echem** to start the program.

   Note: If you are starting the program immediately after installation, you must reboot your computer before starting the program. If you don't, the newly installed GPIB driver will not be loaded.

The program checks the available memory at start-up. It may tell you that you are low on memory but let you continue. If you see the memory-low message, you should exit the software and attempt to free up more conventional memory before running the software. You can free up memory by unloading any unneeded memory-resident utilities (typically loaded through your autoexec.bat file).

When the program is low on conventional memory, some menus will leave blank areas on the screen when you exit them. This might happen, for example, when accessing the help feature. In many places the program will simply redraw the screen. Occasionally a complete screen redraw is a waste of the user's time, so the program will just redraw the current menu on top of any blank areas. This is all normal behavior if your computer is low on free conventional memory when the program starts. If you wish to completely redraw the screen, simply exit the menu displayed and return to the current banner menu. The screen will automatically redraw.

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Menu Navigation

General: With the Model 270 software, you can move between menus or select items within a menu with the mouse or keyboard. Generally, either method is designed to be intuitive, so go ahead and experiment. You'll find that you can figure out how to move to another selection or menu using just the information presented on the screen. Appendix A describes the menu structure and shows the paths to the submenus.

Banner Menus: All operations begin with a selection from a banner menu. A banner menu consists of a row of selection names enclosed in boxes spanning the top of the display screen. All boxes on the banner except the rightmost box contain the names of functions or other banners that you can select. The rightmost box contains the name of the banner.

Pulldown Menus: Most functions are listed on pulldown menus. A pulldown menu is "pulled down" from a banner when you make a selection from the banner. When you're finished with the pulldown menu, you "roll it up" by pressing <Esc> or the space-bar key. It consists of one or two columns of selection names enclosed in boxes dropping vertically from the banner name. The pulldown menus for the techniques are the largest. They consist of two wide columns and encompass almost the entire screen.

Dialog Boxes: Dialog boxes can appear anywhere on the screen, but usually appear in the center of the screen. When they offer additional menu choices, you can select from them in the normal manner. When they ask for text or numbers to be typed in, all editing keys are active (see Editing Keys later in this chapter). If the dialog box requests numerical input, the limits of the parameter will usually be displayed and all input outside this range will be rejected.

Operating with a Mouse

General: Selecting menu options in the Model 270 software with a mouse is easy. You position the arrow cursor on the screen just as you do in other applications - simply move the mouse until the cursor is over the desired position. With the Model 270 software it makes no difference which button you click. In short, to make a selection, simply point and click the mouse.

Alternative Cursor Positioning: You can also use the cursor keys (sometimes called *arrow* or *direction* keys) and Enter key to emulate the mouse. In this method, you press the appropriate cursor keys to position the cursor over the desired selection. Holding a key down for more than a moment speeds up the cursor movement. Pressing Enter makes the selection. (Note that on some keyboards the Enter key is called the Return key. If your keyboard has a Return key, use that when this manual, or a help message, refers to the Enter key).

However, the cursor keys are not as convenient as the mouse. With the mouse, the cursor moves instantaneously and you position the cursor and select without changing your hand position. The cursor and Enter keys may come in handy if your mouse becomes unavailable. An alternative is to use the menu hotkeys (see Operating from the Keyboard on the next page).

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Leaving a Pulldown Menu or Dialog Box: When a pulldown menu is displayed, and you don't wish to make a selection, simply position the cursor outside the menu and click. The pulldown menu will roll up and you can select another item from the displayed banner. If you have clicked on a menu item and change your mind, simply continue to hold the mouse button and drag the cursor off the menu before releasing the mouse button.

The same holds true for any dialog box. When you click outside the box, the box will disappear. You can then select another item from the displayed menu.

Operating from the Keyboard

General: Operating the Model 270 software from the keyboard is straightforward. For a banner, you simply enter the first letter of the selection name. For a pulldown menu or dialog box, you usually enter the first letter of the desired selection. Thus the key you press acts as a hotkey, causing the program to automatically execute that menu option. You don't have to press the Enter (or Return) key.

On some menus, multiple hotkey entries are required to make a selection. You must enter the exact characters displayed on the menu, but you can disregard case. The program uses both alphabetic and number characters in the abbreviations and accepts number input from either the alphanumeric keyboard or numeric keypad. If you use the numeric keypad, you must first activate it by pressing the Num Lock key.

Leaving a Pulldown Menu or Dialog Box: When a pulldown menu is displayed, and you don't wish to make a selection, simply press the space bar or the escape (Esc) key. The pulldown menu will roll up and you can select another item from the displayed banner.

The same holds true for dialog boxes as well, unless you are prompted for text or a number. When you press the space bar or the escape (Esc) key, the box will disappear. You can then select another item from the displayed menu.

The Main Menu: Your Starting Point

The Main menu is the starting point for all operating sessions using the Model 270 software. It appears when you boot the software, type in **echem** at the DOS prompt, and press any key at the Model 270 title screen. It also appears whenever you select Main from another system menu.

The Main menu is a central base of operations. In many cases you must go through the Main menu to get from one banner to another, although the function keys can sometimes take you there directly from one menu to another.

For example, if you've just finished editing and running an experiment and wish to expand the data display, return to the Main menu and select Graph. Then select Zoom, and then Expand, to select the region of the screen to be magnified.

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Main Menu Selections

The following paragraphs provide a brief introduction to the selections on the Main menu. More details are given in Part 2.

File: Displays the File pulldown menu. From there you can save/recall data, print/plot data, or import/export data. You may also shell to DOS, quit the program, or access the cut/paste menu. See Chapter 5 for information on how to use the File pulldown menu. Cut/Paste operations are explained in Chapter 27.

Setup: Selecting Setup sends you to the Setup banner menu. From there you can specify the type of experiment to run, edit the specific parameters for the experiment, and save and recall experiment setups. The Setup menu also allows you to start your experiment. See Chapter 6 for information on how to use the Setup banner menu. Also, Chapter 12 shows you how to edit your setup.

Graph: Selecting Graph takes you to the Graph banner menu. The Graph banner allows you to choose the plot style, integrate your data, overlay plots of different data files, expand or contract or set axes limits of the plot, determine coordinates, and add labels to the plot. See Chapter 7 for information on how to use the Graph banner menu.

Numeric: Selecting Numeric sends you to the Numeric banner menu. From there you can perform numeric operations on your data. You can view/edit the data file point-by-point. Also, you can perform data math allowing you to add/subtract or multiply/divide the data by a constant. File math is also available to allow your to add or subtract other data files. Finally data-smoothing is also available here. See Chapter 8 for information on how to use the Numeric banner menu.

Defaults: Selecting Defaults sends you to the Defaults banner menu. From there you can specify the screen colors, the experimental conditions, and the plotting and file parameters. After you have specified the default settings, you can save them to disk for later recall. See Chapter 9 for information on how to use the Defaults banner menu.

Help: Selecting Help displays the first of the Main Menu Help screens. The messages on these Help screens tell you how to use the Model 270 Help system and describes the Main menu selections. You can also press F1 at any time for help; see Using Help in the next section.

Using Help

Asking for Help: There are two ways of asking for help while you are running the software. Pressing the F1 key or clicking on **Help** will always give you context-sensitive help whether you are selecting from a banner, pulldown, or dialog box, or entering a value in a dialog box. When only a banner is displayed, entering the letter H will also access Help.

Types of Help Screens: The following paragraphs describe the three types of Help screens in the Model 270 software:

- **Banner Menu Help:** The first type is the banner menu Help screen. When

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you select Help from a banner, the screen provides an overview of the functions and operations that can be initiated from the banner. You won't find detailed information on this type of Help screen - just an overview of the menu options available.

You can reach the banner Help screen by pressing the F1 key, clicking on the Help menu selection, or pressing the H key when only the banner is displayed.

- **Pulldown Menu Help:** The second type is the pulldown Help screen. When you "pull down" a menu from a banner and ask for help, the screen provides information about the items on the pulldown, such as how a function works or how parameters on the pulldown interact with each other. The information will be somewhat technical, but not always detailed.

  You can reach this screen by pressing the F1 key when the pulldown menu is displayed.

- **Dialog Box Help:** The third type is the dialog box Help screen. When you select an item from a pulldown menu, a dialog box appears. If you ask for help then, the help message will provide detailed information, such as the allowable range of parameter values or the effect of an entered value on another parameter.

  You can reach this screen by pressing the F1 key when the dialog box is displayed.

Paging through Help: The Help messages are arranged sequentially. You page through the messages by pressing the PgDn (Page Down) key, and you go back to a previous screen by pressing the PgUp (Page Up) key until you're at the desired screen. Or you may click the left or right mouse button to page up or down through the message. Also, you may press <Home> or <End> to go to the first or last page of the Help message.

Exiting Help: To leave a Help screen at any time, simply press the <Esc> key, space bar, or <Enter>. Or, click both mouse buttons simultaneously (not easy!). The program will return you to where you were before you asked for help.

Editing Keys

The editing keys are active whenever you are requested to enter text or numbers. Their functions are listed below.

Home: Moves the cursor to the start of the line.

End: Moves the cursor to the end of the line.

Left: Moves the cursor back one character.

Right: Moves the cursor forward one character.

Backspace: Deletes the character to the left.

Del: Deletes the character at the current cursor location.

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Ins: Toggles between Insert and Overwrite modes. The status of the Insert/Overwrite mode is preserved from entry to entry. That is, it stays in whatever mode you set it to until you change it again.

Down Arrow: Sets the input to the default, or last entered, value. May be used at any time during input.

Ctrl-Enter: Pressing Enter (or Return) while you hold down the Ctrl key deletes to the end of the line.

Enter: Indicates that you have completed your entry. Note that on some keyboards the Enter key is called the Return key. If your keyboard has a Return key, use that when this manual, or a help message, refers to the Enter key.

Esc: Aborts the entry.

F1: Although not strictly an editing key, the F1 key is always available to summon Help messages while you are typing in values or comments.

Function Keys

The Model 270 software uses the function keys to perform special functions. In many cases, a function key provides a shortcut to navigating through several menus to reach your objective. The following paragraphs describe the functions of these special keys:

F1: The F1 key is used to ask for Help in using the Model 270 software. When you press the F1 key, a context-sensitive Help message appears, giving a concise explanation of the function you are using. See the Using Help section on the previous page for more information about the Model 270 Help system.

Shift-F1: Pressing F1 while you hold down the Shift key displays a list of the function key functions.

Ctrl-F1: Pressing F1 while you hold down the Ctrl key displays a list of the editing key functions (see Editing Keys on the previous page).

F2: The F2 key, in most situations, opens the Cut/Paste Setup dialog box. In the Graphics mode, under Curve options, and in the PARCalc, QuickCalc and R_p Calc options on the Analyze menu, it is used to cut/paste values. See Chapter 8 for a detailed description of its use in the Curve mode.

F3: The F3 key displays the numeric values of points on the current plot. It provides a shortcut to selecting Numeric on the Plot banner menu.

F4: The F4 key takes you directly to the Curve pulldown menu on the Graph banner menu.

Alt-F4: Redraw Screen.

F5: The F5 key takes you directly to the Zoom pulldown menu on the Graph banner menu.

F6: The F6 key takes you directly to the Labels pulldown menu on the Graph

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banner menu.

F7: The F7 key takes you directly to the Edit pulldown menu on the Setup banner menu.

Alt-F7: Pressing F7 while you hold down the Alt key executes Run from the Setup banner menu.

Shift-F7: Pressing F7 while you hold down the Shift key takes you directly to the New Technique pulldown menu on the Setup banner menu.

F8: The F8 key takes you directly to the Plots pulldown menu on the Defaults banner menu.

Ctrl-F8: Pressing F8 while you hold down the Ctrl key toggles between current and current density data displays.

Shift-F8: Pressing F8 while you hold down the Shift key toggles through point mode, line mode, and line & point mode data displays.

F9: Pressing F9 causes you to be prompted as if you had selected Save Data from the File pulldown menu on the Main banner menu.

Shift-F9: Pressing F9 while you hold down the Shift key saves your setup.

F10: The F10 key prompts you for a file name for data recall.

Shift-F10: Pressing F10 while you hold down the Shift key causes the program to prompt you for a setup file to recall.

Entering Values

When the Model 270 program requests input, it opens a dialog box. In some cases, you can select one of the displayed values, much as you would select a banner or menu item. In other dialog boxes, you must enter the desired value from the keyboard using either decimal or scientific notation. For example, to enter a parameter value of 3 millivolts from the Edit menu, you can key in either of the following numbers:

.003 (decimal notation)
3E-3 (scientific notation)

Since E in scientific notation represents 10, the second number is equivalent to 3 $\times 10^{-3}$. There is no need to enter the units, as the units used are displayed on the parameter lines.

When a Pass option is displayed for a parameter, you must enter the letter "p" to select it. Also, when you want to enter a potential relative to the open circuit potential instead of the reference electrode potential, you may enter the value with the letter "o" (for "open circuit") before pressing the Enter (or Return) key. The letter "o" may precede or follow the entry, and spaces are allowed. For example, if the above values are specified relative to the open circuit potential, you key in one

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of these lines:

o.003 or 0.003 o (decimal notation)
o3E-3 or 3E-3 o (scientific notation)

Likewise, you may enter certain letters to scale your input. For example, an input of 200m at a prompt for a potential value causes 200 mV or 0.200 V to be entered. The program understands the following abbreviations. These abbreviations, of course, are case sensitive. You must distinguish between capital letters and lower case.

M (mega) = 10^6
k (kilo) = 10^3
m (milli) = 10^{-3}
u (micro) = 10^{-6}
n (nano) = 10^{-9}
p (pico) = 10^{-12}

Note: If you select a dialog box inadvertently and wish to return to the menu to make another selection, simply click outside the box or press Enter without entering a value.

System Checkout and Tutorial

Go through the following procedure to ensure that your entire system is communicating correctly. In doing this you will also begin to become more familiar with using the Model 270 software to run your experiments.

1. Make sure that the system connections are completed and the system is properly booted (refer to Starting the Program on page 13).

2. Type **echem** if you have not already started the Model 270 program.

3. As this is your first session with the Model 270, you need to tell it where to find your potentiostat. So, select Defaults from the Main banner menu.

4. Select Experiment.

5. Select Assign GPIB.

6. Select PSTAT.

7. Select the address that corresponds to your potentiostat address. The factory default is 14, which will work for this initial checkout. If you are using a potentiostat with a different address set on it, select that address.

8. Press <Esc> until you are back at the Defaults banner. You will probably want to save this address from session to session, so select Save on the Defaults banner. Next select Main to return to the Main banner.

9. From the Main banner menu, select Setup.

10. From the Setup banner menu, select New Technique.

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11. From the New Technique pulldown menu, select Linear Sweep Voltammetry.

12. Select COMMENT (CO) on this menu, type in **Initial Checkout** (to identify the type of experiment), and press the Enter (or Return) key.

13. Select SCAN RATE (SR), enter **100** (for 100 mV/s), and press <Enter>.

14. Select FINAL POT. (FP), enter **+1.0** for +1.0 V and press <Enter>.

15. Press the space bar to exit the Setup menu.

16. If you are using a Model 273 or 273A potentiostat, turn the EXT/DUMMY switch on the electrometer to DUMMY. If you are using a Model 173, set the cell selector slide switch to DUMMY CELL. If you are using a VersaStat, hook up a resistor or an experimental cell to the cell leads. If you are using a Model 263, use the System key to toggle to the Function menu. Then set the Cell Type to **Dummy**.

17. Select Run from the Setup banner menu.

    If you are using a Model 273 or 273A potentiostat, the REMOTE indicator will light on the potentiostat, and the LCD panel will display the message MODEL 270 ELECTROCHEMICAL SOFTWARE. On the computer monitor, you will receive the message: "Please enable cell switch." Press the Cell Enable switch on the potentiostat, and then press any key to continue the experiment. The following message will now appear on the monitor: "Really run the dummy cell?" Normally you would be preparing to collect data on your experimental cell, and this prompt reminds you to switch the EXT/DUMMY switch to EXT. Just press the space bar to ignore the message. If you are using the Model 263, just type **Y** to continue and use the dummy cell.

    The TALK and LISTEN indicators will begin flashing (Model 273 or 173/276). On the Model 263, the LED display screen will indicate **Remote** in the upper left-hand corner.

18. The system will collect 501 data points in 10 seconds, the experiment will end, and the After Run banner menu will appear on the screen, indicating that the experiment ran successfully and that the Model 270 Research Electrochemistry Software is communicating properly with the potentiostat.

19. Select Graph to analyse this sample data.

20. Select **Curve** from the Graph banner menu. Then, select **Line Fit**. Type **B** to make the first data point the beginning of a linear least squares analysis of the experimental data. Press the **<End>** key to go to the last data point and then type **E** to make this point the end-point of the linear least squares analsyis. The program will now display the least squares slope and intercept. The units on the slope are A/V and its inverse gives you the resistance in ohms of the dummy cell. For a Model 273A, the slope will the 10.0E-6 A/V, corresponding to a 100 kΩ resistor. For a Model 173/276 or a Model 263, the slope will be 0.1E-3 A/V, corresponding to a 10 kΩ resistor.

21. Press the space bar twice to return to the Graph banner menu.

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22. If you wish to run another experiment at this point, all you need to do is to return to the Setup banner menu (via the Main banner) and select Edit to alter the experiment parameters. Or, you may select another technique from the New Technique pulldown (on the Setup banner) before you edit the experiment parameters.

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Overview

From the File pulldown menu, you can save data to a selected disk, recall a data file from the hard disk, import or export data from or to a floppy disk, or, without ending the Model 270 operating session, exit to DOS to execute a DOS command. You can also view the current setup parameters in the data file, specify data files for cut/paste operations, or quit the program.

For example, if you wanted to save your data after plotting, you would select the Main menu from the Plot menu, and then select the File menu. From the File menu, you would select Save data and respond appropriately to the prompts. The program will write your data onto the specified disk and assign it the specified file name.

To return to the Main menu from the File pulldown, simply press <Esc> or click outside the File pulldown.

Save Data

Definition: Allows you to save the data presently in memory to a specified data file on disk.

Purpose: To save the final results of your experiment. Also, if your data is critical, it's a good idea to save before you perform any editing, graphic, or calculation functions with it, or temporarily exit to DOS. The program will prompt you to save your data in some of these situations.

Procedure: The Model 270 program accommodates all of the DOS conventions for file names. You must enter at least a file name. If you don't enter a disk drive designation, the program will automatically save the data to the drive\directory specified in the Defaults Files Drive\Dir menu option.

Entering a drive designation will override the default and your data will be written to the specified drive. See the description of the Files function on the Defaults banner menu in Chapter 9 for more information about drive, directory, and file name protocol.

Note: You are given the option of saving the data in memory whenever you select a function that irreversibly alters it.

Procedure **Saving the Data in Memory**

Use this procedure to save the data in memory:

1. With data in memory from a measurement or from recalling it from disk, select File from the Main banner menu.

2. Select Save data.

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3. Enter a file name or press Enter if the file name is already displayed. If no file of the same name already exists, the program will write the data from memory into the specified file.

4. If a file of the same name already exists, the program will display the message "Overwrite data on disk?" If you wish to overwrite the data, enter a Y. If not enter an N and specify a different file name.

Get Data

Definition: Allows you to recall data from a specified data file on disk.

Purpose: Lets you recall raw or modified data from disk to view, edit, graphically modify, perform calculations upon, or send to an output device.

Procedure: The Model 270 program accommodates all of the DOS conventions for file names. You must enter at least a file name. If you don't enter a disk drive designation, the program will automatically save the data to the drive\directory specified in the Defaults Files Drive\Dir menu option.

Entering a drive designation will override the default and the program will search for the data on the specified drive. See the description of the Files function on the Defaults banner menu in Chapter 9 for more information about drive, directory, and file name protocol.

Procedure **Recalling Data from a Disk**

Use this procedure to recall a data set from disk:

1. Select File from the Main banner menu.

2. Select Get data.

3. Enter a file name or press Enter if the file name is already displayed.

4. To avoid overwriting critical unsaved data residing in memory, the program will display the question "Save Data?" If you don't want to save it, enter an N. If you want to save the data, enter a Y, save the data, and go through this procedure again.

5. When you answer N to the question, the program loads the specified data set into memory. It will be automatically displayed after the recall has occurred.

Print/Plot Data

Definition: Creates a hardcopy of the plot on a printer or plotter.

Note: Printer output is usually faster than plotter output.

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Use this procedure to send the plot to a printer or plotter:

1. Select Print/Plot from the File Pulldown menu.

2. Select either Graph or Report. If the output device is properly connected, the program will automatically send the plot to the selected device type. If both a printer and a plotter are installed, you will be prompted for the device to use. If you select Report, you will receive a print-out containing only the experimental setup information and the results of any calculations performed.

View Parameters

Definition: View Parameters allows you to view the parameters associated with the data set in memory of the selected cell. When you make this selection, the full set of parameters associated with the data automatically appears on a pulldown menu, while the parameter values for the most recent setup in memory remain intact. You cannot change any of the displayed values while viewing the parameters.

Purpose: When you recall data from memory and plot it, View Parameters allows you to view specific parameter values of the associated setup. This gives you more information to use in interpreting your data. **Note:** View Parameters will display the experimentally measured open-circuit potential if you wish to know that value after your experiment.

Edit Sample

Definition: Experimental parameters associated with potentiostat settings cannot be changed after an experiment. You may wish, however, to alter the comment associated with the data file, the type of reference electrode used, or change the electrode area after the experiment. The Edit Sample menu will allow you access to these parameters.

Purpose: Lets you update the comment for the data file or correct inaccurate information about your experimental sample or reference electrode.

Import Data

Definition: Import Data allows you to bring data into the Model 270 program from simple ASCII text files, including Cut/Paste files. The data in a file to be imported must be in the form of a series of numbers on each line, with one *x,y* data pair on each line.

Before you can import a text file, you must define the fields that contain the *x* and *y* data, and specify the file delimiter (character or space) that separates the fields. You set these parameters from the Files pulldown menu on the Defaults banner menu. See Chapter 10 for details.

The software will stop importing data when it has either processed the whole file

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or encounters a line it cannot process. Then, it will display the number of *x,y* data pairs imported.

Export Data

Definition: Export Data allows you to quickly create text files containing selected data for use by other programs. The data is exported as *x,y* data pairs with one data pair on each line. The *x* value appears first on the line and the *y* value second. The *x* and *y* values are separated by the file delimiter you have specified in the Files pulldown menu on the Defaults banner menu. Refer to Chapter 9 for details.

The values correspond to the currently displayed data style. That is, log (*I*) is output instead of *I* if that is appropriate for the current data style. Also, any deleted points are omitted, as are any points not currently visible on the screen.

You may turn on Autoscaling to output all the data by default, or use the Expand feature to output a subset of the current data. The Autoscale and Expand features are accessed from the Zoom pulldown menu on the Graphics banner menu (see Chapter 8).

Data may also be output either scaled or unscaled as determined by the Export Mode setting in the Files pulldown menu on the Defaults banner menu. If scaled, 1.2 μA of current is output as 1.2. If not scaled, it is shown as 1.2E-6. See Chapter 10 for details of the Export Mode setting.

Cut/Paste

Definition: The Cut/Paste *function* can be activated with the F2 key from any option selected from the Curve pulldown menu, or from any Calc option on the Analyze pulldown. It allows you to save selected portions of the data to a specified file after performing graphics functions on the data in memory. You do the actual graphics and cut/paste operations from the Curve menu using the Free Cursor, Curve Cursor, and Line functions before you press the F2 (Cut/Paste) key. See the description of the Cut/Paste function in Chapter 27 for details.

DOS

Definition: The DOS function lets you exit to the computer disk operating system. From there, you can execute a DOS command or, if sufficient memory is available, run other utility programs without ending the operating session.

Note: Because the Model 270 software uses most of the computer memory, there is a limit to the size of the second program you can run using the DOS function. If you try to run a program that requires more than the available space, the program may run slowly or it may not run at all. In the latter case, the program will display this message:

Program too big to fit in memory

It is a good idea to save your data before exiting to DOS. If you try to run a

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program without sufficient memory and the system "hangs," you could lose your data. You should also avoid running any memory-resident utilities, or "TSRs," as they could also hang the system.

If the Model 270 program does not let you exit to DOS, it is using all of the available memory. If your computer is short of memory, only the printer or the plotter should be installed, not both.

Purpose: The DOS function is useful when you are in the middle of an operation that you don't want to end, but need information or wish to perform a DOS procedure before continuing. Displaying a directory of a selected disk with the DOS DIR command is one example. Another is formatting a blank disk with the Format command so that you can save a data set to it.

Procedure: When you select DOS, the program removes the Model 270 File pulldown menu from the screen and passes control to DOS. When the DOS prompt appears, you can enter any DOS command. It will execute when you press Enter (or Return). To get back to the Model 270 File menu, type the word "exit" and press Enter.

Example **Displaying a Directory**

The following example describes the procedure for temporarily exiting the Model 270 program, displaying the root directory of the C drive, and returning to the Model 270 program:

1. Select Main.

2. Select File.

3. Select DOS. The program automatically exits to the DOS operating system, while retaining the setup and data that you were using. It displays this message before the DOS prompt is displayed:

   Enter "exit" to return to Model 270

4. At the DOS prompt, enter this line:

   dir c:\

   and press Enter.

5. DOS will display the root directory of the C drive (usually the hard disk) and display the DOS prompt again.

6. Enter another DOS command or enter "exit" to return to the Model 270 program. See your DOS manual for information about DOS commands.

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Quit

Purpose: The Quit function is used to leave your experiments, exit the Model 270 program, and return control of your computer to DOS. From there you can run other programs if you wish. You can return to the Model 270 program at any time the DOS prompt is displayed by typing **ECHEM** and <Enter>.

If you have unsaved data or an unsaved experimental setup, the program will ask you whether you want to save them. If you answer "yes" you will be prompted for a file name before you are allowed to exit. If experiments are running, you will be prompted twice before you are allowed to exit. If you exit while experiments are running, your data will be lost!

Pressing the Esc key causes the program to abort the exit process and continue running.

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Overview

From the Setup banner menu, you can edit your experimental setup, select a new measurement technique, recall a setup from disk, save it to disk, or start an experiment. You will often come to the Setup menu from the Main menu to begin a session.

You will also come to the Setup menu after running an experiment. For example, you may determine that the setup is incorrect and wish to change it before doing a new measurement. Or, you may wish to change one parameter value and perform the next of a series of measurements. Because this operating sequence is common, the After Run menu, which is displayed at the completion of a measurement, allows you to select Setup as soon as the measurement is over.

Main

Definition: Selecting Main returns you to the Main menu, where you can select other banners. This is normally the only way to get to another menu.

For example, if you finish editing a setup and wish to change the plotting mode from a smoothed line to discrete points before running an experiment, you would first exit the Setup menu by selecting Main and then Defaults. Then, from the Plots pulldown menu, you would select Line Type, and choose Points.

Edit

Definition: Displays the experimental setup in memory on a pulldown menu so you can edit the settings. The menu displays the parameters for the current setup or, at boot-up, the default setup for the Tafel Plot technique.

If you want to create a setup for a different experimental technique, select New Technique. If you want to edit a setup previously saved on disk, select Get Setup.

See Part 3 of this manual for specific information about how to create and edit a setup.

New Technique

Definition: Pressing this selection displays the New Technique pulldown menu, which contains thirteen selections. In addition, you can perform stripping experiments using several of the techniques, as noted.

Procedure: When you select a new technique, the program automatically brings

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you to the Edit menu and displays the default setup for the selected technique. See the description of the selected technique in Part 2 for setup information.

Technique Sub-Groups: Although the techniques are listed in a single group on the menu, it's helpful to see that they can be divided into convenient sub-groups. The following list shows these sub-groups.

Voltammetry
- Cyclic voltammetry
- Linear sweep voltammetry
- Square wave voltammetry

Note: These three techniques can also be used for stripping experiments.

Polarography/Voltammetry
- Sampled DC
- Differential pulse
- Normal pulse
- Reverse normal pulse

Note 1: These four techniques can also be used for stripping experiments.

Note 2: When you perform these four techniques on a dropping mercury electrode, they are *polarographic* techniques. When you perform them on an electrode other than a dropping mercury electrode, they are *voltammetric* techniques.

Time-based
- Chronoamperometry
- Chronocoulometry
- Chronopotentiometry
- Controlled Potential Coulometry

Utility
- iR Compensation
- Immediate mode
- Autoexecute

Chapters 12 through 24 show you how to apply these techniques. You can also select from this menu the Autoexecute function and Immediate Mode, described in Chapters 26 and 25.

Procedure: When you select a new technique, the program automatically brings you to the Setup pulldown menu and displays the default setup for the selected technique. See the description of the selected technique in Part 3 for setup information.

Get Setup

Definition: Allows you to recall a experimental setup from a specified file on disk. Normally the file will be a setup file, but you may also recall just the setup from a Model 270 data file as well.

Purpose: Lets you edit an existing setup or run an experiment with it.

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Procedure: The Model 270 program accomodates all of the DOS conventions for file names. You must enter at least a file name. If you don't enter a disk drive designation, the program will automatically try to read the setup from the default disk (see the description of the Drive/Directory function in Chapter 9 under the Files section). Entering a drive designation will override the default and the program will read the setup from the specified drive.

Procedure **Getting a Setup from Disk**

Use this procedure to recall a setup from a specified disk:

1. Select Get Setup from the Setup banner menu.

2. Enter a file name or press Enter if the file name is already displayed.

3. To avoid overwriting the setup in memory, the program will display the question "Overwrite unsaved setup?" If you don't want to save it, select Y. If you want to save the existing setup, select N, save the setup, and go through this procedure again.

4. When you select Y, the program reads the specified setup from disk, loads it into memory, and automatically brings you to the Edit menu where the setup is displayed.

Save Setup

Definition: Allows you to save the setup presently in memory to a specified file on disk.

Purpose: To save your experimental settings between operating sessions. If you want to save a setup and don't plan to run and save the entire experiment immediately, you should save the setup before you perform another operation or quit the operating session.

Procedure: The program accomodates all of the DOS conventions for file names. You must enter at least a file name. If you don't enter a disk drive or drive/directory, the program will automatically save the setup to the default disk drive and directory (see the description of the Files function on the Defaults banner menu, Chapter 9). Specifying the drive or drive/directory will override the default and your setup will be written to the specified drive or drive\directory.

Note: The program will give you an opportunity to save the setup in memory whenever you select a function that alters it.

Procedure **Saving the Setup in Memory**

Use this procedure to save the setup in memory:

1. With the setup in memory, select Save Setup from the Setup menu.

2. Enter a file name or press Enter if the file name is already displayed.

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3. If the file already exists, you will be asked to confirm overwriting it.

Run

Definition: Starts an experiment and takes you to the Run banner menu. Refer to Chapter 11 for information on running experiments and the Run banner.

Note: If you are using a Model 173/276 potentiostat, switch the cell selector slide switch to EXT. CELL just before you select the Run command.

Help

Definition: Selecting Help brings up a series of screens that provide an overview of the functions and operations that can be initiated from the Setup banner menu. You can page through the screens with the Page Up and Page Down keys or the left and right mouse buttons. When you have finished reading the Help information you seek, press <Esc> to return to the Setup banner menu.

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Overview

From the Graph menu, you can specify the style of the plot, add labels to it, overlay plots of other data over it, and expand or contract the plot. You can draw straight lines on it, manipulate a free or a curve-bound cursor, analyze your data, and select a data point for cut/paste operations.

Main

Purpose: Selecting Main returns you to the Main menu, where you can quit the operating session or select another menu. In most cases, selecting Main is the only way to get to another menu. However, the Graph menu is an exception to this. From here, you can move directly to the Plot menu by selecting Plot. To go to any other menu, you must first select Main.

Example: If you finish manipulating a plot and wish to edit a setup from a file on disk, you would first exit the Graph menu by selecting Main and then select Setup and Get Setup.

Overlay

Definition: Superimposes a specified curve from disk onto the displayed plot. If autoscaling is on, the program automatically scales the plot to the dimensions needed to completely display all the data. You can overlay up to five plots onto the original data.

Purpose: You use the Overlay function to visually compare curves taken under contrasting experimental conditions. For example, you may want to compare a family of curves taken at different scan rates. The Overlay function allows you to make qualitative comparisons at a glance.

You may use all of the analysis features on the Graph Curve menu to analyze the most recently overlaid data. Use Plot Plot to return to analyzing the original data file.

If you want to superimpose data files with varying *y*-axis offsets, you can achieve this effect by using the Calc menu Offset function. Simply adjust the *y* values of the data to be overlaid and save it under a new name for recalling with the Overlay function.

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Procedure **Overlaying Data**

Use this procedure to overlay one or more data sets onto a plot:

1. Recall the first data set. The data plots automatically.

2. Select Overlay and enter a file name in response to the prompt. A pulldown listing the possible plotting styles for the data set to be overlaid will appear.

3. Select the plotting style. The second data set will automatically overlay the first and the Overlay box will appear on the left side of the screen to allow you to overlay again. If autoscaling is enabled, the program automatically scales the new plot to the dimensions of the first one.

4. Repeat steps 2 and 3, using the Overlay box.

Style

Definition: Specifies the plot style. Each technique has a different selection of styles that reflect the way in which the potential or current is applied to the cell, the data acquisition mode, and the plotting variations related to the technique.

Styles: The following paragraphs describe the plotting scheme for each listed style. The techniques associated with each plotting style are listed after the style name.

Depending on your experimental technique and the actual experimental setup you may be able to display your data in one of several different formats. In some cases it may be possible to only display the data in one way -- the way in which it was collected. The simple styles are:

- **I vs. E:** Available for CV, LSV, SWV, DPP, NPP, RNP and SDC.
- **I or Q vs. t:** Available for CA, CPC and CC.
- **E vs. t:** Only available for CP.

The additional styles are listed below by technique.

Square Wave Voltammetry (SWV)

I_f, I_r, I_f - I_r, I_f & I_r and All vs. E are available. I_f is the forward current and is measured at the end of the first step in the square wave pulse sequence. I_r is the reverse current measured at the end of the second step in the square wave pulse sequence. I_f - I_r is the difference current. I_f and I_r are what is actually measured, but the difference current is very useful for analytical measurements of the concentration of metal ions in solution. All will display all three curves at once, while I_f & I_r displays both I_f and I_r at the same time.

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Chronoamperometry (CA)

I vs $t^{-1/2}$, 1/I vs. $t^{1/2}$, $It^{-1/2}$ vs. log t. These three styles are useful for determining adherence of the data to the Cottrell Equation:

$$i = nFAD^{\frac{1}{2}} * C / (\pi^{\frac{1}{2}} * t^{\frac{1}{2}})$$

where A is area, C is concentration, n is number of electrons transferred, D is the diffusion coefficient, and F is the Faraday constant. All three styles should give straight lines, since only i and t are varying. The slopes can be used to calculate diffusion coefficients if A, C and n are known for a compound. Note that frequently the presence of significant charging currents can cause the plot of $It^{1/2}$ vs. log t to be non-linear.

If you performed a double potential step experiment (E2 and T2 not set to PASS) then there are two additional styles available:

-I(t)/I(t-T1) vs. t/T1 for t > T1 and ***-I(t)/I(T1) vs. t/T1 for t > T1***

These plots are an attempt to compare the current recovered in the second step of the experiment to either the current during the first step of the experiment or the current at the end of the first step. The numbers on both axes range from 0 to 1 and give an indication of the presence and rate of reactions coupled to the electrochemical processes.

Chronocoulometry (CC)

Q vs. $t^{1/2}$ is always available, while Q vs. theta is available for double potential step experiments (E2 and T2 not set to PASS). Q vs. $t^{1/2}$ should be linear according to the integrated version of the Cottrell:

$$Q = 2nFAD^{\frac{1}{2}} Ct^{\frac{1}{2}} / \pi^{\frac{1}{2}}$$

where n, F, A, D and C are defined as before. The intercept is useful in determining the charge due to the double layer and due to adsorbed species, while the slope can be used to calculate a diffusion coefficient.

The Q vs. theta plot is similar, but applies to the second half of the experiment. Q is actually Q(T1) - Q(t) and is plotted vs. $[T1^{1/2} - t^{1/2} + (t - T1)^{1/2}]$. This style is known as an Anson plot and although more complex mathematically it should also give a linear plot and is more useful for studying adsorbed material.

Controlled Potential Coulometry (CPC)

log I vs. t is available. Ideally the current in CPC decays exponentially with time so that this plot should be linear. Deviations from non-linearity can be caused by kinetic complications or by a counter electrode that is too small causing the current to limit at less than its natural value.

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Use this procedure to select a plotting style.

1. From the Main banner menu, select Graph.
2. From the Graph banner menu, select Style. The Style pulldown menu will appear, displaying the styles available for the data set in memory.
3. Select the desired style. The plot will redraw on the screen in the selected style.

Curve

Definition: The Curve pulldown menu offers you several ways to examine your data and display the results in a message box at the top of the screen. You may use the F2 key to cut/paste the information displayed into a previously opened Cut/Paste file.

The Curve Cursor and Free Cursor options allow you to use the mouse or the cursor keys to determine graph coordinates. The Curve Cursor limits you to selecting points on the data plot, while the Free Cursor lets you move anywhere within the boundaries of the graph.

The Area, Peak, and Half-Width options allow you to select a portion of the data to analyze for peak analysis. The Line Fit option lets you select a portion of the data to analyze using a linear least-squares algorithm to determine the slope and intercept. The Draw Line function allows you to draw a freehand line through a portion of the data and display the corresponding slope and intercept. The Erase Line feature removes lines from the screen that were generated with either the Draw Line or Line Fit options. Last, the New Curve option erases all lines, overlays, and any highlighting from the Area, Peak, or Half-Width options.

Note: The Free Cursor, Curve Cursor, and Area, Peak and Half Width options can make their measurements relative to a user-generated baseline, although the default is to make measurements relative to the *x*-axis (see Use Baselines, described in Chapter 9).

You can draw as many as five lines on the screen with the Line Fit and Draw Line routines. These lines are always drawn from one edge of the graph to the other.

Cut/Paste Feature

You may use the F2 key to cut/paste the information displayed into a previously opened Cut/Paste file. See Chapter 27 for a detailed description of Cut/Paste operations.

Curve Cursor

The Curve Cursor lets you use the cursor keys or the mouse to interactively display data coordinates. Generally, moving the mouse or pressing the arrow keys have the same effect. The Page Down key and the Left and Down arrow keys take you lower in point numbers, while Right, Up, and Page Up take you higher in point numbers. The Page Up and Page Down keys move you 25 points at a time, while the others move only 1 point at a time. The Home and End keys take you to the first and last

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data points. You may use the F2 key or type <c> to use the Cut/Paste feature to store the displayed coordinates in the Cut/Paste file. Pressing the spacebar, <Esc>, or <Enter>, or clicking any mouse button, will exit Curve Cursor.

Note that if you have used the Line Fit routine or the Draw Line routine to generate a line on the screen, the Curve Cursor may use one of the lines as a baseline for all values displayed here (see the Use Baselines selection on the Plots pulldown menu, Chapter 9).

Procedure **Manipulating the Curve Cursor**

1. From the Graph menu, select Curve. The Curve pulldown will appear.
2. Select Curve Cursor. A crosshair cursor will appear on the plot and the cursor coordinates will appear at the top of the screen.
3. Use the mouse or the cursor arrows to move the cursor to the desired position.

Free Cursor

The Free Cursor lets you use the cursor keys or the mouse to interactively display graph coordinates without restricting you to the data curve. You may use the F2 key or type <c> to use the Cut/Paste feature to store the displayed coordinates in the Cut/Paste file. If you do so the values are stored with the point number set to -32767. Pressing the spacebar, <Esc>, or <Enter>, or clicking any mouse button, will exit Free Cursor.

Note that if you have used the Line Fit or Draw Line routine to generate lines on the screen, the Free Cursor may use one of the lines as a baseline for all values displayed here (see the Use Baselines selection on the Plots pulldown menu, Chapter 9).

Procedure **Manipulating the Free Cursor**

Use this procedure to manipulate the free cursor:

1. From the Graph menu, select Curve. The Curve pulldown will appear.
2. Select Free Cursor. A crosshair cursor will appear on the plot and the cursor coordinates will appear at the top of the screen.
3. Use the mouse or the cursor arrows to move the cursor to the desired position.

Draw Line

The Draw Line feature allows you to draw straight lines anywhere on the screen. The lines will remain on the plot (unless you delete them with the Erase Line function) until you plot the original data again using the Plot banner Plot function. They will also appear on the printed or plotted output. The Draw Line function can be useful in making quantitative evaluations of the data.

The mouse and the cursor keys can be used to position the crosshair cursor

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anywhere on the graph. Simply position the cursor to the starting point of the line and press <Enter> or click the right mouse button to mark the point. Then, moving the cursor will move the second endpoint of the line to the current cursor position, and will update the message box with the current slope and intercept. The F10 key or the left mouse button is useful to toggle the crosshair cursor between the endpoints so that both can be individually adjusted.

Clicking the right button or pressing Enter a second time exits the Draw Line feature and leaves the line on the screen. Pressing <Esc> or the spacebar will exit Draw Line and erase the line. Note that the lines drawn on the screen with either the Draw Line or the Line Fit feature can be used by the Curve Cursor, Free Cursor, and Area & Peak options as a baseline for measurements (see the Use Baselines selection on the Plots pulldown menu, described in Chapter 9).

You may use the F2 key or type <c> to use the Cut/Paste feature to store the displayed information in the Cut/Paste file. If you do so, the values are stored with the point number set to -32766, the slope is stored as the *x* value, and the intercept is stored as the *y* value. See Chapter 27 for more information.

Procedure **Drawing a Line on the Plot**

Use this procedure to draw a straight line on the plot:

1. From the Graph menu, select Draw Line. An information box will appear above the plot, and a crosshair cursor will appear in the plotting area.

2. Using the mouse or the cursor keys, move the crosshair cursor to where you want the line to begin. Then, press either the right mouse button or <Enter>. This will define the fixed point on the plot through which the line will pass. (The point is shown as a box.)

3. Using the mouse or the cursor keys, move the crosshair cursor away in any direction from the fixed point you have defined. Note that the cursor forms a second point through which the line passes. These two points define the line. By moving the mouse or pressing the cursor keys, you can rotate the line in any direction.

4. By clicking the left mouse button you can reverse the functions of the two points you have defined on the plot. That is, the fixed point will become the movable point, and the movable point will become fixed. Typing the letter <t> has the same effect. Try it both ways.

5. When the line is positioned the way your want it, click the right button or press <Enter> again. This will fix the line on the plot and exit the Draw Line function. If you wish to delete the line, choose Erase Line from the pulldown.

Erase Line

The Erase Line option removes lines from the screen that were generated with either the Draw Line or the Line Fit option. Selecting this function erases only the last line drawn, acting much like an Undo function.

Use this procedure to erase a line you have drawn: With the plot displayed, select

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Erase Line. The program will delete the last drawn line only. If you have more lines to erase, just repeat the process. You can then redraw the lines if you like.

Line Fit

The Line Fit feature allows you to highlight a portion of the data to analyze for a linear least-squares fit. The mouse and the cursor keys can be used to position the crosshair cursor on the data curve in the same manner as the Curve Cursor. The left and right mouse buttons or the <b> and <e> keys can be used to set the beginning and ending points of the selected data. Note that lines drawn on the screen with either the Draw Line or the Line Fit feature can be used by the Curve Cursor, Free Cursor, and Area & Peak options as a baseline for measurements.

The F10 and shift-F10 keys cycle the second line of the message box through several pieces of information. Whenever you update the highlighted section of the data, the display will automatically update with the new slope and intercept. Pressing F10 displays the percentage of relative errors of the slope and intercept. These are the standard deviations resulting from the least-squares calculation expressed as a percentage of the slope and intercept. Also available for display are the current limits of the highlighted section and the correlation coefficient as well as "chi squared" (χ^2). Chi squared is the sum across the data set of the squares of the differences between the calculated *y* values and the actual *y* values. The smaller the chi squared the better the fit.

The correlation coefficient is a more standard statistic for linear least-squares analysis. It varies between 0 and +1 or -1 depending on the sign of the slope of the line. The better the fit the closer the number is to +1 or -1.

You may use the F2 key or type <c> to use the Cut/Paste feature to store any of the displayed information in the Cut/Paste file. See Chapter 27 for more information.

Area, Peak, & Half Width

These features allows you to highlight a portion of the data to determine peak value, integrated area, and full width at half maximum. The mouse and the cursor keys can be used to position the crosshair cursor on the data curve in the same manner as the curve cursor. The left and right mouse buttons or the <b> and <e> keys can be used to set the beginning and ending points of the selected data.

By default, these routines will analyze all of the displayed data to calculate the area, peak, and half-width for you. You may, of course, adjust the endpoints.

Note that if you have used the Line Fit or Draw Line routine to generate lines on the screen, the Area & Peak option may use one of the lines as a baseline for all values displayed here (see the Use Baselines selection on the Plots pulldown menu, Chapter 10). Also note that for scan techniques, the calculated area is in coulombs even though the true area would have units of amps x volts instead of coulombs (amps x seconds). For log plots, the areas determined are always positive.

The F10 and shift-F10 keys cycle the second line of the message box through several pieces of information, including the values of Area, Peak, and Full Width at Half Peak Maximum. Whenever you update the highlighted section of the data, the display will automatically update with the newly calculated quantity. Pressing F10 will allow you to toggle the display to the other types of information.

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You may use the F2 key or type <c> to use the Cut/Paste feature to store any of the displayed information in the Cut/Paste file. See Chapter 27 for more information.

Zoom

Definition: Allows you to graphically expand or contract the plot scale, switch between manual and automatic scaling, and manually set the plot scale.

Purpose: You can use Zoom to focus on selected portions of the curve, especially when you are not interested in a large portion of the data. It also gives you visual control of the plot scale and lets you manually enter precise scaling values.

Expand

The Expand selection lets you zoom in on a selected portion of the plot. This allows you to enlarge selected portions of the curve for closer analysis and to eliminate unwanted portions of the curve.

You can also use Expand in conjunction with the Contract function to visually scale the plot to a desired size. You can do the same thing mathematically, using the Manual function, but you would not have the advantage of seeing the results as you scale the plot.

Procedure **Expanding the Plot**

Use this procedure to expand a portion of the plot to the full size of the plotting area:

1. From the Zoom pulldown menu, select Expand.
2. Position the cursor at one corner of the desired rectangular area.
3. Click the mouse button or press <Enter>. Then drag the mouse until the rectangle covers the desired area. If you change your mind, you can cancel the Expand function by pressing <Esc>.
4. Click the mouse button again or press <Enter>. The data in the selected rectangle will automatically fill the entire screen.

Contract

The Contract selection shrinks the plot by 67% both vertically and horizontally. It does this by increasing the x and y coordinate range by 50% and keeping the plotting area the same. Depending on the current and potential intervals displayed on the contracted plot, the coordinate ranges may be more than doubled.

The result is a plot centered in the screen with approximately one-half the area of the original and blank space outside the contracted plot. This allows you to then use the Expand function to fill the blank area of the screen.

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Procedure **Contracting the Plot**

Use this procedure to contract the plot to approximately one-half of its area:

1. From the Zoom pulldown menu, select Contract.
2. The data will automatically be reduced to one-fourth the area, and will be displayed in the center of the plotting area.

Manual

The Manual function turns off the default Autoscale function and lets you set the plot scale manually by entering high and low values for both the *x*- and *y*-axes. When you select Manual, a pulldown appears, displaying a line for each of the four scaling coordinates. The following paragraphs describe these coordinates:

- **X1:** The X1 coordinate defines the most positive edge of the *x*-axis.
- **X2:** The X2 coordinate defines the most negative edge of the *x*-axis.
- **Y1:** The Y1 coordinate defines the most positive edge of the *y*-axis.
- **Y2:** The Y2 coordinate defines the most negative edge of the *y*-axis.

Procedure **Entering Manual Scale Values**

Use this procedure to enter manual scale values:

1. From the Graph banner menu, select Zoom.
2. Select Manual. The coordinate value lines will appear.
3. Using the mouse or the keyboard, select a line and enter the desired value. The value will appear on the line.
4. Repeat step 3 for each of the lines you wish to change.
5. If you wish to plot with the new scaling values, select the Plot banner menu and then select Plot. The program will draw the plot with the new scaling values.

Autoscale

The Autoscale function is what allows the program to automatically plot the data on the screen when you select Plot on the Plot banner menu. Since Autoscale is the default scaling mode, you don't have to select it to plot. The program automatically determines the smallest possible scale that will encompass all the data points by reading the high and low values of both the *x* and *y* data point coordinates.

Note 1: When you select Manual, the program will deactivate Autoscale until you reselect it. If you quit the Model 270 program, Manual scaling will not be saved as a default setting. Autoscale is always in effect when you start the program.

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Note 2: When manual scaling is in effect, you can change the plotting style. However, you must be aware that changing the style may affect the *x*-axis and *y*-axis scaling values enough to prevent your data from being displayed in the plotting area. Before changing the style, make sure you determine how it will affect the data display.

Labels

Purpose: The Labels pulldown menu allows you to add your own title, *x*-axis, and *y*-axis labels to your graphs. Selecting Edit Title, Edit X Axis, or Edit Y Axis allows you to edit the current labels. You may use the editing keys when entering or modify any of the labels.

The Title + (on)/- (off) option controls whether a user-generated title is displayed. You change the (+) or (-) setting by moving the mouse pointer to the Title line in the pulldown and clicking. Setting Title to (-) causes the screen to be redrawn without a user-generated title. Setting it to (+) causes the screen to be redrawn with the title you have written. The title is displayed centered above the graph. But note that toggling the (+)/(-) setting has no effect until you exit the Labels menu.

Similarly, the Axes + (on)/- (off) option controls whether user-generated labels for the *x*- and *y*-axes are displayed. Setting Axes to (-) causes the screen to be redrawn without axes titles. Setting it to (+) causes the screen to be redrawn with them. Again, toggling the (+)/(-) setting has no effect until you exit the Labels menu.

Note also that the *x*- and *y*-axes do not automatically have *x* and *y* labels. You must enter them manually. The labels are blank by default, so if you turn on the axes labels, you may have to enter text to get an *x*- or *y*-axis label to display. The easiest way to do it is to have Axes set to (-) when entering *x* and *y* labels.

The axes and title labels can be turned on and off without erasing them from the program. The program will automatically store your labels and the status of the (+)/(-) settings with your data when it is saved. Note that this is not automatic, although the program does consider unsaved labels as unsaved data. If you attempt to exit without saving your data (and labels), you will be reminded to save them. Recalling a data file with stored labels will reload the labels and restore their (+)/(-) settings.

On some screens (CGA most commonly), the *y*-axis label may not fit completely and may be truncated depending on the size of the screen font. When this happens, the text is still intact and will be displayed completely if the graph is printed or plotted.

Help

Definition: Selecting Help brings up a series of screens that provide an overview of the functions and operations that can be initiated from the Graph banner menu. You can page through the screens with the Page Up and Page Down keys or the left and right mouse buttons. When you have finished reading the Help information you seek, press <Esc> to return to the Graph banner menu.

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Overview

The Numeric menu allows you to perform a number of operations that change the way your data appears on the screen. The simplest operation available is the Delete function on the View/Edit menu. It allows you to delete single data points from the display. The File Math menu allows you to add or subtract two experimental data sets run under similar conditions. The Data Math menu allows you to perform simple four-function type calculations on your data. You can multiply or divide your data by a constant or add or subtract a constant from your data. Finally the Smooth Data menu gives you the option of applying either a sliding average or a Savtisky-Golay smoothing function to your data.

Caution!

Any time you make global changes to your data values, your data cannot be retrieved. *The sole exception here is the Delete Data function under View/Edit which can be reversed with the Undelete function.* If your data is critical, make sure you save your data *before* performing any manipulations on the Numeric menu. You should also avoid saving the modified data with the same filename, since that would effectively erase the original data from your disk.

Main

Purpose: Selecting Main returns you to the Main menu, where you can quit the operating session or select another menu. Selecting Main is the only way to get from the Calc menu to another banner menu.

View/Edit

Definition: Lists the data on the screen by point number and *x/y* coordinates, and allows you to delete a selected range of points.

Note: Deleting a data point does not remove it from the data set. Your original data is never changed. When you delete a point or points, the data is no longer displayed when you plot the data set. It is used in smoothing, but not in any other calculations. The first column of numbers gives each of the nine displayed data points a selection number (always from 1 to 9). Selecting one of these numbers (from the keyboard or mouse) will delete an active point or undelete a deleted point. Also note that deleting data points alters the results of calculations. ***For this reason, editing your data causes all calculations to be erased.***

The View/Edit pulldown menu appears when you select View/Edit. From this menu you can view the data point values and edit individual data points. The cursor keys can be used with this menu. The <Home> and <End> keys jump to the first and last page of data, respectively. The up and down arrow keys move toward the top or bottom of the data set one point at a time, while <Page Up> and <Page Down> do so one screenful at a time.

When this pulldown menu first appears, it displays the *x* and *y* values of nine data points, beginning with the point specified by the "First point of range" entry on the

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menu. It contains five columns of data point information, including, from left to right, the row number of the nine displayed points, the point number within the data set, the delete marker (blank for undeleted data, an asterisk for deleted data), the *x*-axis value, and the *y*-axis value. The following paragraphs describe the function of each column in detail:

- **Select (Row Selection Number):** The numbers in this column give each of the nine displayed data points a selection number (always from 1 to 9). They do not indicate the point number within the data set. Selecting one of these numbers (from the keyboard or mouse) will delete an active point or undelete a deleted point.

- **Pt# (Data Set Point Number):** The numbers in this column reference the values on a particular row to a point number within the data set. These numbers will change depending on the data range you've selected below. They will always be sequential and no numbers will be skipped, even if you delete the data point.

- **Del? (Delete Marker):** When you delete a data point, the program puts an asterisk in this column. A blank space indicates an active (or undeleted) data point.

- **X:** The value in this column indicates the *x*-axis (or abcissa) value of a particular data point. The value is displayed in scientific notation.

- **Y:** The value in this column indicates the *y*-axis (or ordinate) value of a particular data point. The value is displayed in scientific notation.

Below these nine data points, the menu displays five selectable operations that let you specify the range of data points you wish to view or edit, delete or undelete the specified range, and save selected portions of your data. These menu selections are described below:

- **First Point of Range:** This parameter allows you to enter the first point of the data range you want to view or edit. The default value is 1. If you enter a value, the program will automatically display nine data points beginning with that value. "First Point of Range" is the only value you need to enter if you simply want to view data point values. However, if you want to delete a range of data points, you must also enter a value for the "Last Point of Range" parameter.

- **Last Point of Range:** This parameter allows you to enter the last point of the data range you want to edit. If you simply want to view a range of data points, you don't need to enter a value here. The "First Point of Range" value is enough to display the desired range of points for viewing.

- **Delete Range:** Clicking on this line or entering a "D" will automatically delete the range of data points defined by the "First Point of Range" and "Last Point of Range" parameters. You can confirm the deletion by making sure there is an asterisk (*) next to each deleted data point.

- **Undelete Range:** Clicking on this line or entering a "U" will automatically restore a previously deleted range of data points defined by the "First Point of

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Range" and "Last Point of Range" parameters. You can confirm the undeletion by making sure there is no asterisk (*) next to each restored data point.

- **Cut/Paste Range:** The Cut/Paste function allows you to save selected portions of your data to a specified file after performing graphics functions on the data. You can use the Cut/Paste Range selection to specify the range of the data to be saved. Refer to Chapter 27 for a detailed explanation of Cut/Paste operations.

Procedure **Viewing Data**

Use this procedure to view numerical data beginning with a selected point:

1. Plot the data on the screen using the Plot function.

2. Select Numeric. The Numeric pulldown will appear.

3. Enter a "First Point of Range" value to specify the first data point you want to appear on the screen (1 is the default value). You can use the curve cursor from the Graphics menu to determine the first data point of interest. The program will display nine points of the data set, beginning with the selected first point.

4. To change the first point (and thus display more of the data), select the "First Point of Range" and enter a new first point value (you don't have to view the data sequentially--feel free to skip around). Use this method to scroll or skip through as much of the data as you wish.

Procedure **Editing a Range of Data Points**

Use this procedure to delete or undelete a specified range of data points:

1. Plot the data on the screen using the Plot function. (Once plotted, the data does not have to be plotted again to be listed and edited, even if you leave the Plot menu and return.)

2. Select Numeric. The Numeric pulldown menu will appear.

3. Enter a "First Point of Range" value to specify the first data point of the desired range (1 is the default value). You can use the curve cursor from the Graphics menu to determine the first data point of interest.

4. Enter an "Last Point of Range" value to specify the last data point of the desired range (1 is the default value).

5. Select "Delete Range" or "Undelete Range" on the Numeric pulldown. The program will automatically delete or undelete the specified range of points. The program will display nine points of the data set, beginning with the selected first point. An asterisk (*) should appear to the right of the point number for each deleted point and a blank space for each undeleted point.

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Procedure **Editing Individual Data Points**

Use this procedure to selectively delete or undelete individual data points:

1. Plot the data on the screen using the Plot function.

2. Select Numeric. The Numeric pulldown will appear.

3. Enter a "First Point of Range" value to specify the first data point of the desired range (1 is the default value). You can use the curve cursor from the Graphics menu to determine the first data point of interest. The list of data points will appear.

4. To delete (or undelete), enter the row number of the desired data point or click on it. The program will automatically delete an active point or undelete a deleted point. Note that this will cause any calculation results to be invalidated and therefore erased from the screen.

5. Scroll or skip through the rest of the data set with the or the mouse and edit any further points the same way.

File Math

Definition: Lets you add or subtract another data set from a data set in memory. When you select File Math, you are given a choice between +File and -File and then prompted for the name of the file to add or subtract.

Purpose: These operations allow you, for example, to subtract a cyclic voltammogram of a blank solution from a cyclic voltammogram of your sample solution to remove the background current for your sample voltammogram.

Note: When you use the File Math functions, the program performs a point-by-point addition/subtraction of the two data files. The two data sets must have been collected under almost identical experimental settings for the subtraction to be carried out. The program will refuse to perform the operation if the data sets are not matched closely enough.

If the experiments that produced the two data sets match fairly closely, the program may perform the operation but the results may not be valid. This is because the program may subtract points that do not correspond to each other in some way. The Model 270 software makes some checks to ensure that the subtraction is valid, but you must ensure that the experiments are matched well enough to perform a valid subtraction.

For example, for a subtraction involving two cyclic voltammograms, the program ensures that the technique, the number of points, the acquisition mode, and the initial and final potentials are the same. However, it does not check the scan rate. Thus, if the subtraction involves data sets acquired at two different scan rates, you must be aware of this dissimilarity and its consequences.

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Procedure **Performing a Blank File Subtract**

Use this procedure to create a difference plot:

1. Recall the first CV data file so that it is in memory.

2. From the Numeric menu, select File Math.

3. Select -File.

4. Enter the file name of the second blank CV data set in the dialog box. The program will automatically subtract the current values in the entered file from the current values in memory, and then redraw the graph to display the difference.

Data Math

Definition: Allows you to add/subtract a constant to/from the data or to multiply/divide the data by a constant.

Purpose: The Data Math options can be useful in a variety of ways. For example, you might use /Constant to divide current in a cyclic voltammogram by the sample concentration. This would be useful when carrying out a study of electrochemical behavior as a function of concentration as it will make the experiments easier to overlay and compare.

When prompted for a number, enter any real number, using scientific notation if desired; for example, 0.0014 or 1.4E-3.

Menu Options:

+ **Constant:** Add a constant value to your data.
- **Constant:** Subtract a constant value from your data.
* **Constant:** Multiply your data by a constant.
/ **Constant:** Divide your data by a constant.

Procedure **Rescaling the Data Values**

Use this procedure to rescale your data by a specified factor:

1. Make sure the data of interest is in memory. If you have not just finished making the measurement, recall the data from disk.

2. From the Numeric menu, select Data Math and then /Constant.

3. Select the data type to rescale, Current, Potential, or Time.

4. Enter the rescaling value in the dialog box. The data values will rescale and the screen will redraw automatically.

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Smooth Data

Definition: Lets you apply a Sliding Average or a Savitsky-Golay smoothing algorithm to the potential, current, or time data.

Purpose: Smoothing can reduce the effects of noise or jitter. The most common use of this function is for smoothing current to minimize the effects of high-frequency noise. Smoothing allows you to produce a plot in which the general trends in the data are not obscured by experimental noise.

Procedure: When you select Smooth from the Numeric banner menu, the Smooth pulldown menu appears. This pulldown lets you select either of the two available smoothing techniques: Savitsky-Golay (G), or Sliding Average (A). The following paragraphs describe the two selections on this menu:

Savitsky-Golay: The Savitsky-Golay smoothing method uses a curve-fitting approach based on a linear, quadratic, or cubic function. You select the function you wish to use by pressing L, Q, or C or by clicking on the name of the desired function. You cannot specify the number of points used in the fit—only the type of fit. For a Linear smooth, three points are used; for a Quadratic smooth, five are used; and for Cubic, seven. The three functions are explained below:

- **Linear:** When you select Linear, the program applies a linear least-squares polynomial algorithm to smooth the data. This algorithm replaces the center point in each sequence of three data points with a value calculated from a fit to the linear least-squares equation.

- **Quadratic:** When you select Quadratic, the program replaces the center point in each sequence of five data points with one point calculated from a fit to a quadratic equation.

- **Cubic:** When you select Cubic, the program replaces the center point in each sequence of seven data points with one point calculated from a fit to a cubic equation.

Sliding Average: The Sliding Average technique is fairly simple. In it a number of points is averaged around the current data point and then the averaged value replaces the original data point. The program prompts you for the number of points to use in the average. The entire data set is replaced this way, creating a smaller variation from one point to the next and a smoother graph.

Procedure **Smoothing**

Use this procedure to smooth your data using a specified method:

1. Make sure the data of interest is in memory. If you have not just finished making the measurement, recall the data from disk.
2. From the Numeric menu, select Smooth Data.
3. Select Savitsky-Golay or Sliding Average.
4. Enter the appropriate value for the sliding average or pick the type of Savitsky-

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Golay smooth to perform. The experimental data collected (almost always current) will be smoothed and then automatically redrawn on the screen.

Help

Definition: Selecting Help brings up a series of screens that provide an overview of the functions and operations that can be initiated from the Numeric banner menu. You can page through the screens with the Page Up and Page Down keys or the left and right mouse buttons. When you have finished reading the Help information you seek, press <Esc> to return to the Numeric banner menu.

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Overview

From this menu, you can specify the screen colors, the experimental conditions, and the plotting and file parameters, and save the default settings to disk.

The settings made from this menu are not saved automatically when you quit the program. You must select Save from the Defaults banner after making your selections if you want them to become the default settings each time you use the Model 270 software.

Main

Definition: Selecting Main returns you to the Main menu, where you can select other banners. This is normally the only way to get to another menu.

Example: If you finish specifying the default settings and wish to edit a setup from a file on disk, you would first exit the Defaults menu by selecting Main and then select Setup.

Colors

Definition: Lets you specify colors for the system menus, the highlighted items, the calculated results, the first plot and plot background, and screen overlaid plots. The current colors settings are always marked with an asterisk (*). You can also turn off the use of colors on overlay plots independent of the overlay colors.

The program will not let you select the same color for both background and foreground colors. You can, however, inadvertently set an overlay color to the background color, if you select your overlay colors before you select the plot/background color. In this case the asterisk marking the current overlay color disappears.

Colors Menu: When you select any of the first four options on the Colors pulldown menu, the available color selections appear as a grid of two-digit numbers. Each number is displayed in the two colors it represents, so all you have to do is select the number of the color pair you prefer.

Note: The number of colors displayed on the color selection grid will vary according to the monitor and video adapter you are using. With a VGA monitor or an EGA monitor with 128K or more of memory, you have a choice of 8 colors—or 56 color pairs. With an EGA monitor with less than 128K of memory, you have a choice of 4 colors—or 12 color pairs. With a CGA, you have a choice of two settings—black on white or white on black. With a Hercules-compatible monitor, you also have a choice of two settings—light on dark or dark on light.

The following paragraphs describe the selections on the Colors pulldown menu:

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- **Menu Colors:** This selection allows you to specify a color pair for all system banners, pulldown menus, and dialog boxes. The box color of each grid selection represents the background color of the banner, pulldown menu, or dialog box. The color of the number represents the foreground color.

- **Highlight Colors:** This selection allows you to specify a color pair for all *highlighted* items, error messages, and the Curve pulldown menu message box. A highlighted item is an item that you have selected. The box color of each grid selection represents the background color of the highlight. The color of the number represents the color of the highlight characters.

- **Plot/Background Colors:** This selection allows you to specify the color of the initial plot of a data set and the background for all system screens. The plot and background colors are not directly related and are paired here only for convenience of selection. The background color of each grid selection represents the system screen color. The number color represents the color of the initial plot. To differentiate plot overlays, the program automatically assigns a new color to each plot overlaid onto the initial plot.

- **Results Color:** The Results color selection determines the color of the results on all graphs. Simply enter the two-digit code corresponding to the foreground color you would like. Note that you cannot select the background color, since it is determined by the Plot/Background Colors setting.

- **Screen Overlay Colors:** Screen overlay colors allows you to specify the individual colors used for each of as many as five overlay files. When you select this option, a dialog box with five overlay selections appears. If you are using only one overlay, select 1st Overlay from this box. Then, enter the two-digit code corresponding to the foreground color you would like for the overlay. If you are going to overlay another plot, select 2nd Overlay and repeat the process. Note that you cannot select the background color, since it is determined by the Plot/Background Colors setting.

- **Use Overlay Colors:** Use Overlay Colors allows you to turn colors for an overlay off quickly without having to reset each individual value for all your overlays. Use overlay colors also determines whether color is used on plotter output. If set to Yes, the axes are drawn on the plotter with pen 1, the first data file with pen 2, the second with pen 3, and so on. If set to No, all plotted output is drawn with pen 1.

Note: If you want your new selections to become the default settings each time you use the Model 270 software, select Save from the Defaults banner menu.

Experiment

Definition: This selection allows you to control various experimental conditions that affect how your measurements are taken and how the results are displayed. You can change the display potential reference, choose the scan rate units, specify how the cell is turned on and off during an experiment and whether the experiment will use any of the potentiostat's triggering capabilities, and whether the potentiostat is reset before each experiment. You can configure the software for multiple potentiostats and assign the cells to them.

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Scan Rate

Selecting Scan Rate opens a menu that lets you choose the unit of measurement for your scan rate setting. You may choose between mV/s, and V/s.

Triggers

The Triggers setting controls whether the potentiostat waits for a trigger before starting the experiment, or generates a trigger when starting the experiment.

The Auxiliary Interface connector on the rear panel of the Model 273 and Model 273A potentiostats has both a Trigger In and a Trigger Out line. Trigger In delays the start of a measurement until pin 2 of this connector becomes active low (0 V). Trigger Out causes the potentiostat to output a logic 0 (0 V) pulse at pin 4 at the start of each data acquisition period. You can use these lines to automatically control the potentiostat or an external device at the start of an experiment. See the descriptions of the TRIG and WFT commands in your Model 273 or 273A manual for more information.

Triggers Menu: When you select Triggers, a menu appears. The following paragraphs describe the selections on the Triggers menu:

- **None:** Selecting None deactivates all triggering.

- **Out:** Selecting Out causes the potentiostat to output a Trigger Out pulse at the beginning of each data acquisition period. For a polarography experiment, the potentiostat would send a new pulse every time it dispense a new drop. If you wish to use an electrode other than the EG&G PARC Model 303A, you could use these pulses to automatically dispense new drops. The Trigger Out line can be connected to an external device to automatically control its operation.

  Example: In a chronoamperometry (CA) experiment, you may want to measure the spectral changes as a function of time as the CA data is being you taken. In this case, you can connect the Trigger Out line to a rapid-scanning spectrophotometer. This will automatically synchronize the CA current monitoring with spectrophotometer data collection.

- **In:** Selecting In causes the potentiostat to wait for a trigger before starting the experiment. The pulse must be a logic 0 (0 V). Do not select this option unless you are sure an external trigger will be sent to the potentiostat. If it isn't, the program will hang up waiting for the potentiostat to finish the experiment.

  The Trigger In line can be connected to an external device to automatically control the beginning of a measurement.

  Example: You can connect a hand-held switch to the Trigger In line to signal the potentiostat that you have just injected a sample. This will avoid wasting time when you're using a sample that changes rapidly when it is introduced into a solution.

- **Both:** Selecting Both activates both the Trigger In and Trigger Out lines.

Cell Control

The Cell Control setting determines whether the Model 270 automatically turns the cell on and off for during the experiment. The program will always turn the cell on if it is off at the start of the experiment, otherwise no useful data would be collected. If Automatic is selected, the program will turn off the cell at the end of the experiment. If Manual control is selected, it is left on at the end of the

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experiment. Automatic is the most convenient, but Manual gives the operator greater control. The following paragraphs describe the two methods of cell control:

- **Automatic:** If you select automatic cell control, the program will turn the cell on at the beginning of the experiment and off at the end of the experiment.

  Also, the program will automatically dislodge and dispense drops, purge the solution, and turn the stirrer on and off under control of your setup.

- **Manual:** If you select manual cell control, the program will turn the cell on at the beginning of the experiment, but will not turn it off at the end of the experiment.

  In manual mode, the program will not automatically perform a dislodge/dispense operation, purge the solution, or turn the stirrer on and off. You must use Immediate mode to perform these operations. See the description of Immediate mode in Chapter 25.

Reset Pstat

The Reset Pstat (potentiostat) function allows you to activate or deactivate automatic reset of the potentiostat at the start of each session. If set to Yes, the program automatically resets the potentiostat when it first accesses it by sending it a DCL (Device Clear) command (a part of EG&G PARC's Electrochemical Command Set). Thus, your potentiostat will start Model 270 operations from a known reset condition whenever you boot. This is the recommended setting. If you set Reset Pstat to No, then old instrumental settings will persist during your experiments whether you want them or not.

Assign GPIB

The Assign GPIB selection is used only if you have changed the original factory GPIB address setting of 14 on your potentiostat. First set the address on the potentiostat (refer to Appendix C) and then enter the address for the potentiostat on this menu.

Note: If you want your new selections to become the default settings each time you use the Model 270 software, select Save from the Defaults banner menu.

Plots

Definition: This menu lets you specify how your graphs are to be displayed on the screen, printer, or plotter. You can specify the current and potential plotting conventions and whether to plot current densities instead of currents. You may also choose whether to display the data in a line drawing mode or as individual points. Furthermore, you can specify whether to have a list of the setup parameters included with the plot when you output it to a printer or plotter.

Setup Output

The Setup Output option allows you to choose whether the program will list the setup values with the plot when it is sent to the output device. Furthermore, you can have these parameters shown in either complete or abbreviated form.

When you select Setup Output, a dialog box with the words None, Short, and Long appears. Selecting either Short or Long will cause the setup to be listed on a hard-copy plot of *nonoverlaid* data. Note that these parameters are never output for overlaid data, as each overlaid file has its own experimental parameters that can vary.

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If Long is selected, 40-character fields are used consistent with the display on the Edit menu. The Short setting gives an abbreviated display of the experimental parameters (22-character fields with 2-letter parameter abbreviations).

Selecting None will tell the program not to list the setup with the plot. This will give you less-cluttered graphs for publication-quality figures.

Line Type

The Line Type selection allows you to choose the line type used to plot the data. The following paragraphs describe the two selections:

- **Points:** This selection tells the program to use a small square to represent each point on the curve.

- **Lines:** This selection tells the program to use straight lines to connect the data point coordinates on the curve. The points themselves are not represented, so at high resolutions or in a contracted view of the plot, the curve appears as a continuous line.

- **Both:** This selection tells the program to plot points and draw lines.

  At low resolutions (with a CGA monitor, for example) and in an expanded view, the lines appear jagged and the vertical portions of the curve can appear as dots. The breaks in a low-resolution line occur at the data point coordinates, but they may not all be visible.

  For overlaid data, the selection of line or point style is determined automatically by the program.

Use Baselines

If you have drawn several baselines with the Draw Line or Line Fit functions, the Use Baselines option lets you choose which line to use as the baseline for the Free Cursor, Curve Cursor, and Area, Peak, & Half Width functions. The options are None, First Line (the first one you drew), and Last Line (the last one you drew). The default setting is None.

Current Density

The Current Density option lets you choose whether to display the current as current density. The default setting is to display it as current density. Note that the Sample Area must be changed from the default setting of 1 cm^2 to see any difference in the graph.

Cathodic current

This selection allows you to choose the cathodic (reductive) current orientation. The following paragraphs describe the four selections:

- **+ Up:** This selection tells the program to plot the data so that cathodic current is positive and a positive-going (cathodic) current moves upward.

- **- Up:** This selection tells the program to plot the data so that cathodic current is negative and a negative-going (cathodic) current moves upward.

- **+ Down:** This selection tells the program to plot the data so that cathodic current is positive and a positive-going (cathodic) current moves downward.

- **- Down:** This selection tells the program to plot the data so that cathodic current is negative and a negative-going (cathodic) current moves downward.

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Potential

Allows you to choose the potential orientation. The following paragraphs describe the two selections:

- **Left is +:** Causes a positive-going potential to be plotted from right to left.
- **Right is +:** Causes a positive-going potential to be plotted from left to right.

Reference Potential

When you specify the reference electrode in an experimental setup, you would normally plot the resulting data using the same reference. However, there may be times when you do not want the plot to be referenced to the same electrode as the experimental reference electrode. The Plot Ref setting lets you display the data relative to a reference electrode different from the one used to collect the data. For example, if you specified SCE as the reference when you collected the data, you can choose to display the data relative to an NHE electrode here. In that case the potential values are offset by the potential difference between the two reference electrodes. For example, 0.5 V vs. SCE becomes 0.7415 V vs. NHE.

Note that the original data is not altered by the choice of a plot reference electrode. In all cases, the data is stored relative to the Setup reference electrode.

Reference Potential Menu: When you select Reference Potential, a menu appears from which you may select one of several different reference electrode settings. You may select from six electrode types, a user-specified reference potential, and a Pass option. The following paragraphs describe these choices:

- **Pass:** The Pass setting causes the data to always be displayed relative to the original reference electrode. This is the default selection for the plotting reference. Selecting Pass tells the program not to convert the original reference electrode potential to a new one when you plot the data. Press P to make this selection.
- **User:** This selection converts the plotting reference to a user-entered potential. Press U to make this selection. You will be prompted to enter a potential.
- **SCE:** This selection converts the plotting reference to the potential of a saturated calomel electrode (SCE). An SCE reference electrode has a potential of 0.242 V versus a normal hydrogen electrode. Press S to make this selection.
- **SSCE:** This selection converts the plotting reference to the potential of a sodium saturated calomel electrode (SSCE). An SSCE reference electrode has a potential of 0.236 V versus a normal hydrogen electrode. Press 2 to make this selection.
- **Ag/AgCl:** This selection converts the plotting reference to the potential of a 1 M KCl/saturated silver chloride electrode. This type of reference electrode has a potential of 0.222 V versus a normal hydrogen electrode. Press A to make this selection.
- **NHE:** This selection converts the plotting reference to the potential of a normal hydrogen electrode. A hydrogen reference electrode has a potential of 0.000 V and is the accepted standard for other reference electrodes. Press N to make

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this selection.

- **Hg/HgSO4:** This selection converts the plotting reference to the potential of a mercury/mercurous sulfate electrode. A mercury/mercurous sulfate reference electrode has a potential of 0.616 V versus a normal hydrogen electrode. Press H to make this selection.

- **Cu/CuSO4:** This selection converts the plotting reference to the potential of a copper/copper sulfate electrode. A saturated copper/copper sulfate reference electrode has a potential of 0.30 V versus a normal hydrogen electrode. Press C to make this selection.

Files

Definition: The Files menu lets you select a number of formatting options for your data files, and specify the drive and directory in which they are located.

Drive\Dir

The Drive\Dir selection allows you to specify the default drive and directory for the Model 270 data files. The program uses the entered drive\directory for file operations (any file search preceding a file read or write operation). The drive\directory specification is commonly known as the *path*.

The entered drive\directory becomes the default path for the data files until you specify a new one. You can override the default at any time simply by specifying a path when you enter the file name. If you do not specify a path, the program simply uses the default drive\directory when performing operations with that file.

Use the standard DOS file name protocol and restrictions to enter the drive and directory. See your DOS manual for details, or use F1 to ask for Help when entering a file name.

Note: To use the DOS default drive\directory setting for the data files, select Drive\Dir and enter .\ (period backslash). This cryptic code is DOS' way of designating the current directory. The DOS default drive and directory are the ones that were active when you started the Model 270 software. If you used the batch file created by the installation program, your current directory will be the Model 270 program directory.

File Delimiter

The File Delimiter option allows you to choose the delimiter character that will be used to separate, or delimit, values in the files. You may choose the delimiter from several ASCII characters.

The file delimiter affects data import, data export, and the Cut/Paste files. For data export, the *x* and *y* values output are separated by this character. In Cut/Paste files, the five fields are separated from one another by this character. For data import, the import data file is expected to have its various fields separated by this character.

When you select File Delimiter, a menu appears from which you can choose the comma, semicolon, space, or tab character as the delimiter. The default setting is the comma, which will suffice for almost all applications.

Export Mode

The Export Mode option determines whether exported data is scaled or unscaled. If you select Scaled, 1.2 μA of current is output as 1.2. If you choose Unscaled, it

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is output as 1.2E-6.

X Import Field #
Y Import Field #

The X and Y Import Field # settings control how input fields in an import data file are mapped to the program's *x* and *y* data values. Using these settings you may assign *x* and *y* to be any of the fields on a line in the import file. The separating character between fields is determined by the File Delimiter setting.

If you select No Field for a field number, the program will store 1 through the total number of points in that field. The program assumes that each line of the import data file contains only data for one data pair, and that there are enough fields to actually access the fields specified by these settings.

Save Mode

The Save Mode allows you to select either the ASCII or the binary format for storing data on disk. Binary files are smaller and load faster, but ASCII files can be used to import data into other programs such as a spreadsheet. You can easily edit or examine your data in a word processor if the files are in ASCII format. The same is not true of a data file stored in binary format. The following paragraphs describe these two formats:

- **ASCII:** This is the acronym for the American Standard Code for Information Interchange. When ASCII-formatted data is displayed by some application programs (such as the DOS TYPE command), the data appears as human-readable text. For this reason, ASCII files are sometimes referred to as *text* or *straight* text files, and are useful for interchange with other programs.

- **Binary:** This data format packs the data more tightly than a data point transferred in ASCII code. This allows for faster data transmission and results in smaller disk files.

Note 1: The header portion of a data file is *always* ASCII-formatted. The data can be either binary or ASCII, depending on what you selected. See Appendix B for more information.

Note 2: ***Never*** use an ASCII text editor to edit a binary file. You could lose all of your data.

Note 3: If you want your new selections to become the default settings each time you use the Model 270 software, select Save from the Defaults banner menu.

Save

Definition: Lets you save all of the selections you made from the Defaults menu during your last session into a special file (DEFAULTS.270). When you next run the Model 270 software, these values will automatically be loaded and take effect.

Purpose: To allow you to customize the wake-up state of the Model 270 software.

Procedure: To save the default values, simply select Save on the Defaults banner menu. The program will automatically save the default settings to the DEFAULTS.270 file. The next time you boot the software, this file will be used to set up the system defaults.

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If you ever wish to return to the preset program defaults, copy the DEFAULTS.270 file from your original distribution diskette into the directory on your hard disk where you keep your Model 270 program files (normally the M270 subdirectory).

Help

Definition: Selecting Help brings up a series of screens that provide an overview of the functions and operations that can be initiated from the Defaults banner menu. You can page through the screens with the Page Up and Page Down keys or the left and right mouse buttons. When you have finished reading the Help information you seek, press <Esc> to return to the Defaults banner menu.

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10 Chapter Ten Run Menu

Overview

The Run menu appears when you press the Run key from the Setup Banner. It allows you to retain control of the measurement during the run. You can stop the measurement at any time, advance the measurement to the next stage, clear the plotting area on the screen, initiate a pause, and later restart the measurement.

Stop

Definition: Pressing the Stop key aborts the entire experiment at any point. The data taken up to the point where you stop the experiment remains intact, so you can still plot and save it.

Purpose: Stop is most often used to abort an improperly designed experiment or to end a measurement when you determine from viewing the runtime plot that you have all the data you need.

Advance

Definition: Pressing Advance moves the programmed scan to the next stage of the measurement. A stage is defined as one of the programmed steps of the experiment, including the purge, conditioning, deposition, equilibration, and data acquisition. Also, if you select Advance during a vertex delay, the program will cancel the remainder of the delay and begin the next part of the measurement.

Purpose: To move to the next stage when you determine that the current stage of the measurement is unnecessary.

Example: If you decide that the purge portion of the experiment is unnecessarily long, you can press Advance to move directly to data acquisition. If another preparatory operation, such as an equilibration, has been programmed, you would move to that operation before going to data acquisition.

Note: Pressing the Advance key also restarts the measurement if you have pressed the Hold key.

Clear

Definition: Clears the runtime plot from the screen. The program will resume displaying the runtime plot from the point at which you pressed Clear as soon as there is enough data to display a significant portion of the plot.

Purpose: To remove data of no interest so that you can view only the data of

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interest during the measurement. For example, you may want to clear the screen after each cycle of a multi-cycle cyclic voltammetry experiment. This will isolate each cycle for easier viewing.

Hold

Definition: Initiates a pause in the experiment. Pressing Advance resumes the experiment where it was interrupted. You can initiate a pause with the Hold selection at any time during the experiment, including data acquisition.

Purpose: Hold gives you manual control of the experiment. It allows you to create a convenient pause when you need to perform another task or wait for a time before continuing.

After Run Menu

Definition: This menu appears after the measurement has successfully run to completion or has been manually stopped. It allows you to initiate a typical post-run operation, such as saving the data, plotting it, or editing the setup to rerun the experiment.

Purpose: All of the selections on this menu, Main, File, Graph, Setup, and Help, are also located on other menus. They are displayed on the After Run menu for your convenience. Pressing any of the selections on this menu brings you to the menu where the function is normally located. See the section describing the normal menu location of the function for more information.

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11 Chapter Eleven Help Selection

Overview

Selecting Help displays the first of the Main Menu Help screens. The messages on these Help screens tell you how to use the Model 352 Help system and describe the Main menu selections.

Using Help

When you select Help from the Main banner menu, the Help screens that appear provide an overview of the functions and operations that can be started from the Main banner. You won't find detailed information on this type of Help screen - just an overview of the menu options available. More detailed Help messages are available from the pulldown menus and dialog boxes. Those messages carry information that is more specific to the operation you are performing when you access them.

Asking for Help: You can reach the Main banner menu Help screens by pressing the F1 key or the H key, as well as by clicking on the Help selection on the banner.

F1 Key: Help is always available on any banner, pulldown, or dialog box by pressing F1.

Paging through Help: The Help messages are arranged in the same order as the Main menu selections. You page through the messages by pressing the PgDn (Page Down) key, and you go back to a previous screen by pressing the PgUp (Page Up) key until you're at the desired screen. Or you may click the left or right mouse button to page up or down through the message. Also, you may press <Home> or <End> to go to the first or last page of the Help message.

Exiting Help: To leave the Help screen at any time, simply press the <Esc> key, the space bar, or <Enter>. Or, click both mouse buttons simultaneously (not easy!). The program will return you to the Main banner menu.

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12 Chapter Twelve Editing a Setup

Overview

The Edit pulldown menu is reached from the Setup banner menu. When you select Edit from the Setup banner, the program displays the setup in memory on a pulldown menu. Since some of the parameter values may already be acceptable for the intended experiment, you can create a new method or edit an old one without having to enter a value for each parameter.

The Edit menu automatically appears when you select Edit, when you select a new technique from the New Technique menu, or when you select Get Setup and specify a setup file. At boot-up, the Edit menu displays the default Tafel plot setup; during operations, it displays the most recent setup.

If you're at the Edit menu and need to create a setup for a technique other than the one displayed, go back to the Setup menu and select New Technique. Note that changing the technique causes the experimental parameters to be automatically reset to the default values. If you wish to temporarily change your technique, use Save Setup to store your setup on disk. If you want to edit a setup previously saved on disk, select Get Setup from the Setup menu.

The original setup is not erased from the disk when you edit a setup. So, if you make a mistake, the error is not permanent. If you have not saved your editing with Save Setup, you may recall the original setup from the disk by selecting Get Setup, and correct the error.

At the upper right of the menu for each technique, a message is always displayed, indicating whether or not the current setup has been saved. Check this message regularly and save when appropriate to ensure that you don't lose critical setup information.

The parameter ranges permitted by the Model 270 software reflect the capabilites of the potentiostat used to control the experiment. They are not software limitations.

Parameter Entry

To enter a parameter value from the Edit menu, first select the parameter by clicking on it or entering the two-character code corresponding to the parameter. This code is displayed to the right of the parameter name on the menu. In the dialog box that appears, you can either select one of the displayed values, or enter the desired value from the keyboard using either decimal or scientific notation. For example, to enter a parameter value of 10 millivolts from the Edit menu, you can key in either of the following numbers:

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0.01 (decimal notation)
1E-2 (scientific notation)

Since E in scientific notation represents 10, the second number is equivalent to 1 × 10^{-2}. There is no need to enter the units, since you enter all values in SI units, which are displayed on the parameter line. (**SI** stands for the International System of Units.)

You may also enter certain letters to scale your input. Continuing the earlier example, an input of 10m at a prompt for a potential value causes 10 mV or 0.01 V to be entered. The program understands the following abbreviations. These abbreviations are case sensitive, of course; otherwise the program could not distinguish M(ega) from m(illi). In other words, you must distinguish between capital letters and lower case.

M (mega) = 10^{6}
k (kilo) = 10^{3}
m (milli) = 10^{-3}
u (micro) = 10^{-6}
n (nano) = 10^{-9}
p (pico) = 10^{-12}

Likewise, when a Pass option is displayed for a parameter, you must enter the letter "p" to select it. (This p overrides the pico scale factor.) Also, when you want to enter a potential relative to the open circuit potential instead of the reference electrode potential, you may enter the value with the letter "o" (for "open circuit") before pressing the Enter (or Return) key. The letter "o" may precede or follow the entry, and spaces are allowed. For example, if the above values are specified relative to the open circuit potential, you key in one of these lines:

o.010 or 0.010 o (decimal notation)
o1.0E-2 or 1.0E-2 o (scientific notation)

Note: If you select a dialog box inadvertently and wish to return to the menu to make another selection, simply click outside the box or press Enter without entering a value.

Parameter Corrections

When you enter a parameter value, the program automatically checks the value against the potentiostat capabilities and other related parameter values. If the entered value is not a multiple consistent with the potentiostat capabilities or the other values, the program may make small corrections.

For example, if you enter a value of 1 V for the final potential in a Linear scan experiment, the program will check the value against the scan increment value. If you have entered a scan increment of .003 V, the program will determine that 1 is not an exact multiple of .003 (the nearest multiple), correct the final potential to

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.999 V, and display it on the Edit menu.

Parameter Interactions

Certain parameter values determine or limit the values for other parameters. When this happens, the parameter value presently being entered becomes an independent variable and the previously entered or default value becomes a dependent variable.

For example, scan rate, scan increment, and step time all interact. If you change the scan rate, the program updates the step time consistent with the present scan increment setting. Likewise, if you change the step time, the scan rate will be modified. See the descriptions of the individual parameters for more information.

Parameter Conflicts

Entering certain parameter values may force related parameter to change to values outside the range allowed by the potentiostat you are using, making it impossible to run the experiment. If this happens, an error message will appear on the screen. You should either change the entered parameter (the independent variable) or change the value of the related parameter (the dependent variable). If it is unclear what parameter is causing the trouble, use F1 to get context sensitive help on the error message.

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13 Chapter Thirteen Common Parameters

Overview

This chapter describes the parameters common to all or most of the measuring techniques you can use with the Model 352 SoftCorr software. You can use these parameters to specify the speed of transition between one step and the next of the applied waveform, define the current range, select iR compensation, filter the cell noise, and select the reference and working electrodes. These parameters function the same way for all techniques.

The potentials applied during conditioning are in relation to the reference electrode or the open-circuit potential. You can specify reference or open circuit when you enter the potential (see "Entering Values" in Chapter 4).

Comment (CO)

This parameter is intended to hold any text a user desires to enter up to 78 characters in length.

If you select Comment on the Setup Edit Menu, you are only changing the experimental setup. If you want to change the comment associated with the data after the experiment has run, select Comment on the File Edit Sample menu.

Condition Potential (CP)

Range: ±8.000 V

Default: 0.000 V

Discussion: Conditioning of the electrode occurs before the start of the experiment and occurs with the cell on and if you are using a 303A the stirrer is on and the purge off. You may specify both the time and potential used. If the Condition Time is PASS (0), the Condition Potential is ignored. Alternatively, you may enter 'p' or 'P' to set Condition Potential to PASS.

Condition Time (CT)

Range: 0 to 32,767 s

Default: 0 = PASS

Discussion: Conditioning of the electrode occurs before the start of the experiment and occurs with the cell on and if you are using a 303A the stirrer is on and the purge off. You may specify both the time and potential used. If the Condition Potential is PASS, the Condition Time is ignored.

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Current Range (CR)

Options:

263, 273/273A:	1 nA to 1 A, Auto
Versastat, 173 + 276:	1 μA to 1 A, Auto

Default: Auto or 1 mA.

Discussion: The current range determines the maximum amount of current that can be measured in the experiment and the resolution of the values measured. The maximum current is 2 x current range and the resolution is current range ÷ 1000. Selecting auto enables autoranging for the potentiostat in which the current range is adjusted to keep the current between 0.15 and 1.90 of the current range.

Autoranging is not available for chronocoulometry or the IR measurement technique. In chronocoulometry, the current range directly affects the charge measurments and they would not be accurate if autoranged. In the pulse techniques, SWV, DPP, NP and RNP, autoranging should be avoided as inaccurate difference currents can result from its use. For IR compensation measurments, the uncompensated resistance maximum and resolution are directly related to the current range. Thus, autoranging could affect the accuracy of the IR measurement. Also note that for this reason that you should avoid turning autoranging on if you have the IR parameter set to Entered.

The 10 nA and 1 nA current ranges available on the Model 263, 273 & 273A potentiostats are generated by automatically using current amplifier gain switches present in the potentiostat. For the 1 nA current range, these switches limits the current resolution to 2 pA and the maximum current to 4 nA. ***Note that autoranging does not include the 1 or 10 nA current ranges. If your currents are this low, you must specify a fixed current range.***

Density

Definition: This parameter is the density of the sample. The density is required only if the Sample Area has been entered and you intend to calculate the corrosion rate (MPY) after running the experiment. It can also be entered after running the experiment and before calculations (see Chapter 21).

Default: 0 g/ml

Deposition Potential (DP)
(Chronopotentiometry Only)

Range: Pass, ±8 V

Default: Pass

Discussion: The Deposition Potential is only used for chronopotentiometry. In all other techniques, the Initial Potential is used as the deposition potential. Deposition occurs before the experiment with the cell on, purge off and the stirrer activated. You may enter 'p' or 'P' for PASS to omit deposition. This is unneeded if the

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Deposition Time is set to 0 or PASS.

Deposition Time (DT)

Range: 0 to 32,767 s

Default: 0 = PASS

Discussion: The Deposition Time setting controls how long a deposition potential is applied to the cell with the stirrer on before an experiment. For chronopotentiometry, there is a separate Deposition Potential, but for all other techniques the Initial Potential is used. Deposition or preconcentration is useful in stripping techniques to take advantage of the fact that some electroactive species precipitate after a redox process. The most common use is for lower detection limits of metal ions when determination are performed with a mercury drop electrode.

Electrode Area (AR)

Definition: The size of the electrode surface area in cm^2. This parameter is required for current density calcu-lations. If you choose not to enter the area during the setup, you may enter it after the experiment has completed by using the Edit Parameters function on the Analyze pulldown menu (see Chapter 21).

Range: 1×10^{-15} cm^2 to 1×10^{15} cm^2

Default: 1 cm^2

Equilibration Time (ET)

Range: 0 to 32,767 s

Default: 15 s

Discussion: The Equilibration Time controls a variable delay during which the cell is on, the purge and the stirrer off. Equilibration occurs at the Initial Potential for all techniques except chronopotentiometry which uses the open circuit or rest potential.

Filter (FL)

Options: Off
I or E 5.3 Hz
I or E 590 Hz (Models 263, 273/273A only)
I/E Filter (Model 263 only)

Default: None

Discussion: The filter setting allows you to turn on a variety of filters depending

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on your potentiostat and the type of data being collected in the experimental technique selected. The most common options displayed are Off, I 5.3 Hz and I 590 Hz. These filters are low-pass cut-off filters and help reduce the effects of high frequency noise during the experiment. This is a useful feature when making measurments at very low current levels, e.g. experiments with microelectrodes at slow scan rates.

If your potentiostat is a Model 263, you can also access a filter in the I/E current measurement circuitry that adds a 10 nF capacitor across the current measuring range resistor. The cut-off frequency of this low-pass filter is dependent on the magnitude of the range resistor and ranges from 1.5 Hz on the 100 nA current range to approximately 5 kHz on the 1 mA and higher current ranges. See the Model 263 potentiostat manuals for more information.

IR Mode (IR)

Options: None, Entered, Measured

Default: None

Discussion: Your experimental solution has a finite solution resistance associated with it and this is present between the reference and the working electrode. This solution resistance couples with the cell current to produce an iR potential drop that keeps the working electrode from being at the programmed potential.

The IR mode setting combines with the Uncompensated Resistance setting (RU) to allow you to adjust the applied potential values to correct for this problem using the positive feedback feature of the potentiostat. You may set this option to None for no compensation or Entered if you wish to use compensation. In that case you can use the RU parameter to enter an estimate of the solution resistance. As an alternative you may use the IR measurement technique to have the program estimate the solution resistance for you. When in the IR measurement technique, a third option of Measured is available which allows you to run an experiment to estimate RU for you. When you return to your original experimental setup the IR mode switches to Entered and the measured RU value is preserved.

Note that the RU value measured and applied is specific to the selected current range. Thus, you should avoid setting the current range to Auto when IR mode is set to Entered.

Line Sync

Definition: This function lets you determine whether or not data acquisition will be synchronized with the power line frequency. Line synchronization is desirable when you use a low-current technique, where noise reduction is critical. The function is available only when you are using a Model 263, 273 or 273A as your potentiostat.

Choices: Yes or No

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Default: No

Purge Time (PT)

Range: 0 to 32,767 s

Default: 240 s

Discussion: The 270/250 software is capable of controlling the purge feature of the model 303A SMDE when properly interfaced to the Model 263, 273/273A potentiostats. This setting controls how long the purge feature of the 303A is enabled. A setting of 0 s is equivalent to skipping the purge and displays as PASS. The cell is normally off during the purge and the 303A stirrer is turned on.

Reference Electrode (RE)

Options: SCE, Ag/AgCl, NHE, Hg/HgSO4, User

Default: User

Discussion: The Reference Electrode setting combined with the Reference Potential setting on the Defaults menu lets you display the data relative to a reference electrode different from the one used to collect the data. For example, if you specify SCE as the reference here before the experiment, you can choose to display the data relative to an NHE electrode later by setting the Reference Potential to NHE. In that case the potential values are offset by the potential difference between the 2 reference electrodes, ie 0.5 V versus SCE becomes 0.7415 V versus NHE. You may select from 7 different reference electrode settings:

Electrode	Potential(V)
SCE	0.242
SSCE	0.236
Ag/AgCl	0.222 (1 M KCl)
NHE	0.000
Hg/HgSO4	0.616
CuCuSO4	0.300
User	entered

You may always set the Reference Potential to PASS to cause the data to be displayed relative to the original reference electrode. The user setting allows you to enter your own values.

Note that if you select Ref. Elec. on the Setup Edit Menu, you are only changing the experimental setup parameters. To change the reference electrode setting after the experiment has been run, select Ref. Elec. on the File Edit Sample menu.

Rise Time (RT)

Options: High Stability & High Speed (Model 263 and Model 273/A only)

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Default: High Stability

Discussion: The Rise Time parameter changes the bandwidth characteristics of the potentiostat. The bandwidth of the potentiostat is a measure of how quickly the control ampflier can respond to a rapidly changing applied potential without degrading the signal. In effect it determines whether or not the potentiostat can keep up.

In the High Speed mode, the potentiostat has a higher bandwidth than in the High Stability mode which translates into a minimization of the amount of time taken for the potentiostat to apply a given potential. In both cases this time is much less than the timescale of data acquisition. In addition the High Speed mode has a disadvantage in that it can cause the potential to oscillate with some cells. This oscillation will look like a ringing effect on current measurement and is most noticeably when either the current range changes while autoranging or the scan direction reverses. With High Stability oscillations are less likely to occur, but still possible. The type of cells that are most prone to show oscillations are those with high impedance reference electrodes. The impedance of your reference electrode depends primarily on its design but can also be affected the precipation of any salts at its tip. Precipation is a common problem when using a saturated aqueous reference electrode in a non-aqueous solution.

Normally this parameter is set to High Stability which is the recommended setting and will suffice for most experiments. None of the techniques in the software require potentials that change rapidly enough to encounter problems with the potentiostat bandwidth. In fact, this parameter is really included for completeness in controlling the potentiostat rather than having major utility. The High Speed setting could be useful in cases where the High Stability mode gives some ringing due to oscillations. In some cases, changing to the High Speed mode may not increase oscillations and might actually minimize the amount of time the potential oscillates. To re-emphasize, this parameter should, in general, be set to High Stability.

Working Electrode (WE)

Options: HMDE, Solid, Rotating or DME

Default: HMDE: 263, 273/273A
Solid: Versastat, 173 + 276

Discussion: This option allows you to specify the type of working electrode used in your experiment. Note only the Solid option is supported for the Versastat & the Model 173 + Model 276 potentiostats due to hardware limitations.

HMDE and DME options specify that you are using a EG&G PARC Model 303/A mercury electrode. If this electrode is properly cabled to a Model 263, 273 or 273A potentiostat, the program can control the electrode's stir, purge and dispense/dislodge features. If you select the HMDE option, the 303A must be set to HMDE or SMDE mode and then program automatically carries out a dislodge and dispense cycle before each experiment. In this case the whole experiment is performed with one mercurcy drop. If you select the DME option and the set the 303A to SMDE or DME, the program carries out a dislodge and dispense operation at the beginning of every sampling period. With the 303A set to DME, the drop

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continues to grow throughout the measurement, while with the SMDE setting the drop is quickly dispensed and does not grow continually during the measurement. The SMDE setting is the recommended setting for the 303A as it is compatible with both the HMDE and DME menu options.

The Rotating option indicates that you are using the EG&G PARC 616 Rotating Disk Electrode (RDE). If selected, the program will try to control the speed of the rotating electrode via the Output BNC on the front panel of a 263, 273 or 273A potentiostat. This BNC is connected to both the 1V/4k and common inputs on the 616 using a BNC to double banana cable.

The Solid option simply indicates to the program that you are using an electrode which does not require any software control.

Uncompensated Resistance (UR)

Range: Varies with Current Range

Default: 0 ohms

Current Range	Resistance Range	Resistance Resolution
1 A	2 Ω	1 mΩ
100 mA	20 Ω	10 mΩ
10 mA	200 Ω	100 mΩ
1 mA	2 kΩ	1 Ω
100 μA	20 kΩ	10 Ω
10 μA	200 kΩ	100 Ω
1 μA	2 MΩ	1 kΩ
100 nA	20 MΩ	10 kΩ

Discussion: This parameter allows you to enter an estimate for the Uncompensated Resistance between the reference and working electrodes. This uncompensated resistance couples with the cell current to introduce a potential drop which keeps the working electrode potential from assuming the programmed value. The value entered here is used to adjust the applied potentials to compensate for this problem. Compensation does not occur unless the IR mode parameter is set to Entered. You may use the IR measurement technique to have the program estimate the uncompenstated resistance for you. In that case the value displayed is the maximum uncompensated resistance that the program can apply given the settings of compensation level and undershoot is less than or equal to the solution resistance multiplied by the compensation level.

Note that the range of the resistance and its resolution is directly related to the current range. Do not set the IR mode to Entered with autoranging of the current enabled. It can affect the accuracy of your data.

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14 Chapter Fourteen Cyclic Voltammetry (CV) Setup

Overview

This chapter describes the parameters unique to a Cyclic Voltammetry (CV) experiment. These parameters define the CV waveform. For additional setup information, refer to these chapters before or during the CV setup:

Chapter 6 . . . . . setup menu
Chapter 13 . . . . . common parameters

Cyclic Voltammetry Defined

In this technique, the potentiostat applies a linear sweep of a programmed potential range which reverses at some point in potential and monitors the current as a function of applied potential.

A typical response curve is similar to that obtained from Linear Sweep Voltammetry, ie. sigmoidal with peaks for electroactive species. The major difference is that Linear Sweep usually observes only Oxidations or only Reductions, while Cyclic Voltammetry can involve both, so that both positive and negative peaks can be observed.

You may specify up to 2 vertex potentials, Vertex 1 (V1) and Vertex 2 (V2) in addition to the Initial Potential (IP), and the Final Potential (FP). In many cases the Final Potential is the same as the Initial Potential and by default the software locks the two together, i.e. if you change IP then FP is updated. You may unlock them by simply changing the Final Potential. It is also possible to set V2 to pass, if you only want one vertex. You may use the Number of Cycles (NC) parameter to specify how many times the potential program is applied, although the program can only store 2 cycles of data. ***If you perform multiple cycles, you should keep the Final Potential set to the same value as the Initial Potential, as otherwise the potential will step radically from FP to IP at the end of each cycle. This will result in an unwanted current spike in the middle of your cyclic voltammogram.***

Traditionally, this technique is performed using a straight analog ramp. Due to the digital nature of the potentiostat, however, the actual ramp applied consists of a series of small potential steps that approximate the linear ramp desired. You may use the Acquistion Mode setting to control where the current is sampled during each potential step, ie. 1/4, 2/4, 3/4 or 4/4 of ST. You should set AM to 4/4 if you intend to use the Model 271 Cool Kinetic Analysis System.

Each of the steps has a height, the Scan Increment (SI) and a width, the Step Time (ST). The Scan Rate (SR) is directly related to these two parameters by SR = SI/ST. You may enter either SR or SI & ST. If you change SR or ST, the program will update the other one by assuming a constant SI value.

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Figure 14-1 shows the applied potential waveform with the experimental parameters labelled.

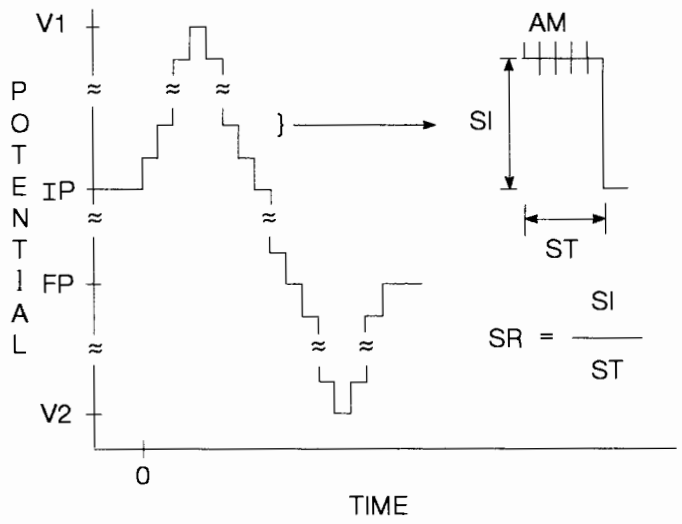


Figure 14-1. CYCLIC VOLTAMMETRY APPLIED VOLTAGE WAVEFORM AND PARAMETERS

Acquisition Mode (AM)

Options: 1/4, 2/4, 3/4, 4/4, All, Ramp

Default: 4/4

Discussion: The Acquisition Mode determines how many times the current is sampled in relation to the Step Time in CV and LSV and also which of those current samples are kept. In addition, it can affect how the Scan Increment is applied.

Before discussing all the nitty gritty details of the Acquisition Mode, there are 3 important points. For the fastest Scan Rates, you will need to use the 4/4 mode and any mode other than 4/4 and Ramp can decrease the maximum Number of Points due to the added current values and memory limitations of the potentiostat. Note that, if you intend to analyze your data with the Model 271 Cool Kinetic Analysis System, you should set AM to 4/4.

Now back to what Acquisition mode really accomplishes. In the simplest mode 4/4, the current is sampled just once at the end of the Step Time. In the 1/4, 2/4 and 3/4 modes the current is sampled various intervals corresponding to 1/4, 2/4 or 3/4 of the Step Time. In these modes, only 1 current values is stored by the potentiostat for later recall by the program. The All mode causes the current to be measured at all 4 intervals during the Step Time and all 4 values to be stored. The All mode decreases the maximum Number of Points that can be collected to 750 (3000 max ÷ 4). The Ramp mode causes the current to be sampled at the end of the Step Time like the 4/4 mode, but changes how the Scan Increment is applied. In all other modes the Scan Increment is just applied in one step at the beginning

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of the Step Time. In the Ramp mode this step is broken up into finer incremental steps as small as 0.25 mV if possible.

Why bother with all of this you ask? A reasonable question. The answer relates to the difference between linear scans generated with true analog ramps or digital ramps in which the ramp is actually a series of small steps. A number of papers have been published on just this topic. To summarize the results of these papers, if your electractive species is electrochemically reversible (properly called Abelian) then a sampling point of 1/4 the Step Time (AM = 1/4) gives a voltammogram most like that produced with an analog ramp. If your system is not electrochemically reversible (Non-Abelian) then a sampling point of 2/4 yields a voltammogram most like an analog ramp. If you don't know whether the system is electrochemically reversible, the All mode saves the day. The Ramp mode is an alternative that keeps the potential steps as fine as possible with the idea the smaller the steps the more the voltammogram will look like that of an analog ramp. Which acquisition mode is right for you? Only you can answer that, but the 4/4 mode is the fastest, is required by COOL, and is in many respects the simplest choice. Ramp mode is a good default and the All mode is a safe choice to see the effect of the varying the sampling point as you can use the Style menu to display the various curves after the experiment.

Final Potential (FP)

Range:
263, 273/273A: ±10.000 V
Versastat, 173 + 276: ±8.000 V
Note: Total scan range must be < 4.0V.

Default: 0.000 V

Discussion: The Final Potential is the potential at which the scan ends. The value is stored with a resolution of 1 mV and may be entered versus the open circuit potential by adding an 'o' or 'O' character to the end of the value.

The value you enter is corrected to the nearest multiple consistent with the scan increment and initial potential. When you change FP you are also changing the total potential range of the scan which combines with the Scan Increment to determine the Number of Points parameter. Thus, it is possible to get a "Too many points" error message after changing any of your potential limits.

Note also that the Final Potential can be locked to the Initial Potential (IP) if they have the same value. When they are locked together, changing IP causes FP to be updated. This is done as a convenience, since many cyclic voltammograms are run with identical starting and ending potentials. You can unlock FP by simply changing its values to something different from IP.

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Initial Potential (IP)

Range:

263, 273/273A: ±10.000 V
Versastat, 173 + 276: ±8.000 V

Default: 0.000 V

Discussion: The Initial Potential is the potential that is applied at the start of the experiment and during the Equilibration Time. Values are stored with a resolution of 1mV and the total potential range must be no more than 4 V. Otherwise a "Potentials out of range" error message will result. Also the total potential range during a scanning technique combines with the Scan Increment to determine Number of Points parameter. Thus adjusting the Initial Potential can also lead to a "Too many points" error message. You may enter the value vs. the open circuit potential by simply adding an 'o' or 'O' character at the end of the value.

The Final Potential (FP) can be locked to IP in CV experiments. What that means is that if IP is the same as FP and you change IP, then FP is updated to the same value. This is done to reflect that in these experiments it is very common to have the last potential applied to the cell be the same as the initial potential. You can unlock FP by simply changing its value to something different from IP.

Number of Cycles (NC)

Range: SC to 32,767 cycles

Default: 1

Discussion: This setting controls the number of potential cycles performed in cyclic voltammetry. Note that multiple cycles are displayed on the screen, but the data set will contain only 2 of these cycles due to memory limitations of the potentiostat. The last cycle is always preserved while the Store Cycle (SC) setting specifies the other cycle to save.

Note that the program will not let you set NC to less than the SC parameter as that would lead to a parameter conflict. Increasing NC will increase the number of points collected during the experiment and if set too high will result in a "Too many points" error message.

Number of Points (NP)

Range: 1 to 3000 points

Default: 200

Discussion: The Number of Points(NP) is the total number of points taken during the experiment. This parameter is not user settable for this technique. Its value is

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shown for informational purposes only. It is controlled by the total potential range of the scan combined with the setting of the Scan Increment.

Note after you change NP, the program adjusts the Time per Point (TP) by dividing the total experiment time by the newly entered number of points less 10 points and correcting TP to the nearest multiple consistent allowed potentiostat values for the TP parameter.

Scan Increment (SI)

Range: 1 to 20 mV

Default: 2 mV

Discussion: The Scan Increment determines how much the potential changes between two data points in the experiment. When combined with the potential limits it determines the Number of Points parameter. When combined with the Step Time for the scan techniques it determines the Scan Rate by following equation:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

After changing the Scan Increment, the program will adjust the values of the Number of Points and the Step Time (except for Square Wave where this parameter is irrelevant). Thus, you may encounter either a "Too many points" error message if the Number of Points exceeds. This limit is normally 3000 but can go as low as 750 in LSV and CV depending on the Acquisition Mode setting. The Step Time setting is adjusted based on the current Scan Rate and may generate a "Step time too short" error message.

Scan Rate (SR)

P'stat:	Model 273/273A	Model 263	Versastat and Model 173 + Model 276
Minimum	1E-6 V/s	1E-6 V/s	1E-6 V/s
Maximum			
Calculated	400 V/s	166.7 V/s	72 V/s
Recommended	160 V/s	66.7 V/s	29 V/s
Default:	20E-3 V/s	20E-3 V/s	20E-3 V/s

Discussion: The scan rate controls how quickly the potential changes in the voltammetric techniques. It is directly related to the scan increment and the step time:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

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After the scan rate is changed, the program updates the step time using the values for scan rate and scan increment.

The maximum scan rates allowed are different from the maximum values recommended. The maximum calculated values assume Scan Increment is set to its maximum value (20 mV), but the resolution of the data is very low. The recommended maximum scan rate is based on a Scan Increment of 8 mV. Note that for lower values of Scan Increment, the maximum Scan Rate you can enter and not get an error message is lowered. For example if the scan increment is 2mV (the default), the maximum scan rate for this Scan Increment is 40 V/s and entering any scan rate over 40 V/s will lead to a "Step time too short" error message. Obtaining higher scan rates requires adjusting the Scan Increment. In addition to adjusting the Scan Increment, you may need to adjust two other parameters, the Current Range setting and the Acquisition Mode, to get higher scan rates. Any Acquisition Mode other than 4/4 slows down the potentiostat. Also setting the Current Range to Auto slows down the potentiostat as well. To get the highest scan rates you must run the experiment on a fixed current range. By changing one or both of these settings you should be able to get rid of the "Step time too short" error message.

Step Time (ST)

Range:

273/273A:	50 μs to 1074 s
263:	120 μs to 1074 s
Versastat, 173 + 276:	275 μs to 1,074 s

Default: 0.1 s

Discussion: The Step Time setting determines how long each Scan Increment is applied. The Step Time is related to the Scan Rate by:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

Although the program will not let you enter a Step Time shorter than the minimum value, you may still encounter a "Step Time too short" error message. There are two parameter that slow down the potentiostat and thus affect the minimum Step Time. Autoranging of current requires extra time, so a setting of Auto for the Current Range can cause a Step Time too short message. The Acquisition mode in LSV and CV also can affect the speed of the potentiostat. Use AM set to 4/4 for the shortest step times.

Store Cycle (SC)

Range: 1 to NC

Default: 1

Discussion: This Number of Cycles (NC) parameter controls the number of

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potential cycles performed in cyclic voltammetry. Although multiple cycles are displayed on the screen, the data set contains only 2 of these cycles due to memory limitations of the potentiostat. The last cycle is always preserved while the Store Cycle(SC) setting specifies the other cycle to save. Note that the program will not let you set SC to greater than the NC parameter as that would lead to a parameter conflict.

Vertex 1 Potential (V1)

Range:
263, 273/273A: ±10.000 V
Versastat, 173 + 276: ±8.000 V
Note: Total scan range must be < 4.0V.

Default: -1.000 V

Discussion: The Vertex 1 Potential is the ending potential of the first segment of the cyclic voltammogram. After the scan reaches V1 it turns toward either Vertex 2 or the Final Potential (V2 = pass). The value is stored with a resolution of 1 mV and may be entered versus the open circuit potential by adding an 'o' or 'O' character to the end of the value.

The value you enter is corrected to the nearest multiple consistent with the Scan Increment and the Initial Potential. When you change V1 you are also changing the total potential range of the scan which combines with the Scan Increment to determine the Number of Points parameter. Thus, it is possible to get a "Too many points" error message after changing any of your potential limits.

Vertex 2 Potential (V2)

Range:
263, 273/273A: ±10 V
Versastat, 173 + 276: ±8 V
Note: Total scan range must be < 4.0 V.

Default: Pass

Discussion: The potential at which the second segment of the scan ends and the scan turns toward the final potential. Enter a value in volts in multiples of 0.001 V or enter 'p' to set to PASS.

As for all potential settings in the experiment, the whole potential range must be under 4 V. If you enter values with more than a 4 V range, you will get a "Potentials out of range" error message.

In addition the total scan range combined with the Scan Increment dictates the Number of Points collected during an experiment. If your settings exceeds the maximum number of points you will be get a "Too many points" error message.

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Vertex Delay (VD)

Range: Pass, 0 to 32,767 s

Default: Pass

Discussion: The delay interval between the first and second half-cycles of the scan, entered in seconds (s). Begins when the scan reaches the vertex 1 potential.

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15 Chapter Fifteen Linear Scan (LS) Setup

Overview

This chapter describes the parameters unique to a Linear Scan (LS) experiment. These parameters define the LS waveform. For additional setup information, refer to these chapters before or during the LS setup:

Chapter 6 . . . . . general menu
Chapter 13 . . . . . common parameters

Linear Scan Defined

In this technique, the potentiostat sweeps the cell potential from an Initial Potential (IP) to a Final Potential (FP) and measures the current that results. A typical response curve is sigmoidal with peaks superimposed. The peaks correspond to electroactive species present in solution or on the electrode surface.

Traditionally, this technique is performed using a straight analog ramp. Due to the digital nature of the potentiostat, however, the actual ramp applied consists of a series of small potential steps that approximate the linear ramp desired. Each of the steps has a height, the Scan Increment (SI) and a width, the Step Time (ST). The Scan Rate (SR) is directly related to these two parameters by SR = SI/ST. You may enter either SR or SI & ST. If you change SR or ST, the program will update the other one by assuming a constant SI value.

Figure 15-1 is a drawing of the applied potential waveform with the experimental parameters labelled:

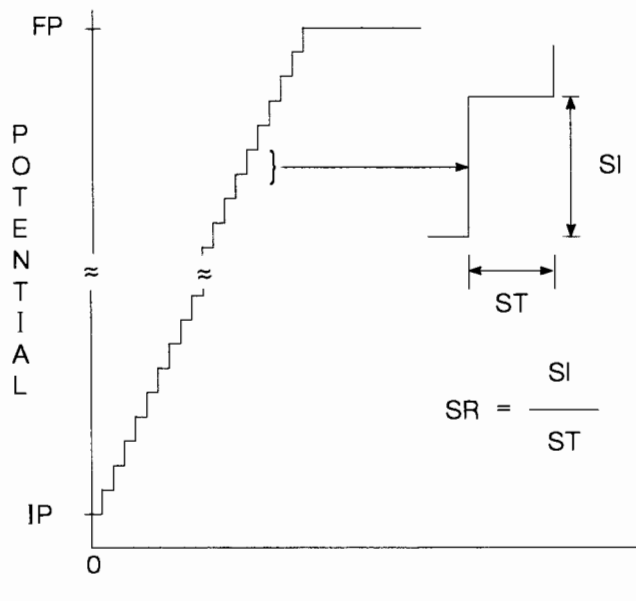


Figure 15-1. LINEAR SWEEP VOLTAMMETRY
APPLIED VOLTAGE WAVEFORM AND PARAMETERS

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Acquisition Mode (AM)

Options: 1/4, 2/4, 3/4, 4/4, All, Ramp

Default: 4/4

Discussion: The Acquisition Mode determines how many times the current is sampled in relation to the Step Time in CV and LSV and also which of those current samples are kept. In addition, it can affect how the Scan Increment is applied.

Before discussing all the nitty gritty details of the Acquisition Mode, there are 3 important points. For the fastest Scan Rates, you will need to use the 4/4 mode and any mode other than 4/4 and Ramp can decrease the maximum Number of Points due to the added current values and memory limitations of the potentiostat. If you intend to analyze your data with the Model 271 Cool Kinetic Analysis System, you should set AM to 4/4.

Now back to what Acquisition mode really accomplishes. In the simplest mode 4/4, the current is sampled just once at the end of the Step Time. In the 1/4, 2/4 and 3/4 modes the current is sampled various intervals corresponding to 1/4, 2/4 or 3/4 of the Step Time. In these modes, only 1 current values is stored by the potentiostat for later recall by the program. The All mode causes the current to be measured at all 4 intervals during the Step Time and all 4 values to be stored. The All mode decreases the maximum Number of Points that can be collected to 750 (3000 max ÷ 4). The Ramp mode causes the current to be sampled at the end of the Step Time like the 4/4 mode, but changes how the Scan Increment is applied. In all other modes the Scan Increment is just applied in one step at the beginning of the Step Time. In the Ramp mode this step is broken up into finer incremental steps as small as 0.25 mV if possible.

Why bother with all of this you ask? A reasonable question. The answer relates to the difference between linear scans generated with true analog ramps or digital ramps in which the ramp is actually a series of small steps. A number of papers have been published on just this topic. To summarize the results of these papers, if your electractive species is electrochemically reversible (properly called Abelian) then a sampling point of 1/4 the Step Time (AM = 1/4) gives a voltammogram most like that produced with an analog ramp. If your system is not electrochemically reversible (Non-Abelian) then a sampling point of 2/4 yields a voltammogram most like an analog ramp. If you don't know whether the system is electrochemically reversible, the All mode saves the day. The Ramp mode is an alternative that keeps the potential steps as fine as possible with the idea the smaller the steps the more the voltammogram will look like that of an analog ramp. Which acquisition mode is right for you? Only you can answer that, but the 4/4 mode is the fastest, is required by the COOL software, and is in many respects the simplest choice.

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Final Potential (FP)

Range:
263, 273/273A ±10.000 V
Versastat, 173 + 276±8.000 V
Note: Total scan range must be < 4.0 V.

Default: -1.000 V

Discussion: The Final Potential is the potential at which the scan ends. The value is stored with a resolution of 1 mV and may be entered versus the open circuit potential by adding an 'o' or 'O' character to the end of the value.

The value you enter is corrected to the nearest multiple consistent with the scan increment and initial potential. When you change FP you are also changing the total potential range of the scan which combines with the Scan Increment to determine the Number of Points parameter. Thus, it is possible to get a "Too many points" error message after changing any of your potential limits.

Initial Potential (IP)

Range:

263, 273/273A: ±10.000 V
Versastat, 173 + 276±8.000 V

Default: 0.000 V

Discussion: The Initial Potential is the potential that is applied at the start of the experiment and during Equilibration Time. Values are stored with a resolution of 1mV and the total potential range must be no more than 4 V. Otherwise a "Potentials out of range" error message will result. Also the total potential range during a scanning technique combines with the Scan Increment to determine Number of Points parameter. Thus adjusting the Initial Potential can also lead to a "Too many points" error message. You may enter the value vs. the open circuit potential by simply adding an 'o' or 'O' character at the end of the value.

Number of Points (NP)

Range: 1 to 3000 points

Default: 200

Discussion: The Number of Points(NP) is the total number of points taken during the experiment. This parameter is not user settable for this technique. Its value is shown for informational purposes only. It is controlled by the total potential range of the scan combined with the setting of the Scan Increment.

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Scan Increment (SI)

Range: 1 to 20 mV

Default: 2 mV

Discussion: The Scan Increment determines how much the potential changes between two data points in the experiment. When combined with the potential limits it determines the Number of Points parameter. When combined with the Step Time for the scan techniques it determines the Scan Rate by following equation:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

After changing the Scan Increment, the program will adjust the values of the Number of Points and the Step Time (except for Square Wave where this parameter is irrelevant). Thus, you may encounter either a "Too many points" error message if the Number of Points exceeds. This limit is normally 3000 but can go as low as 750 in LSV and CV depending on the Acquisition Mode setting. The Step Time setting is adjusted based on the current Scan Rate and may generate a "Step time too short" error message.

Scan Rate (SR)

P'stat:	Model 273/273A	Model 263	Versastat and Model 173 + Model 276
Minimum	1E-6 V/s	1E-6 V/s	1E-6 V/s
Maximum			
Calculated	400 V/s	166.7 V/s	72 V/s
Recommended	160 V/s	66.7 V/s	29 V/s
Default:	20E-3 V/s	20E-3 V/s	20E-3 V/s

Discussion: The scan rate controls how quickly the potential changes in the voltammetric techniques. It is directly related to the scan increment and the step time:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

After the scan rate is changed, the program updates the step time using the values for scan rate and scan increment.

The maximum scan rates allowed are different from the maximum values recommended. The maximum calculated values assume Scan Increment is set to its maximum value (20 mV), but the resolution of the data is very low. The recommended maximum scan rate is based on a Scan Increment of 8 mV. Note that for lower values of Scan Increment, the maximum Scan Rate you can enter and not get an error message is lowered. For example if the scan increment is 2mV (the

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default), the maximum scan rate for this Scan Increment is 40 V/s and entering any scan rate over 40 V/s will lead to a "Step time too short" error message. Obtaining higher scan rates requires adjusting the Scan Increment. In addition to adjusting the Scan Increment, you may need to adjust two other parameters, the Current Range setting and the Acquisition Mode, to get higher scan rates. Any Acquisition Mode other than 4/4 slows down the potentiostat. Also setting the Current Range to Auto slows down the potentiostat as well. To get the highest scan rates you must run the experiment on a fixed current range. By changing one or both of these settings you should be able to get rid of the "Step time too short" error message.

Step Time (ST)

Range:

273/273A:	50 μs to 1074 s
263:	120 μs to 1074 s
Versastat, 173 + 276:	275 μs to 1,074 s

Default: 0.1 s

Discussion: The Step Time setting determines how long each Scan Increment is applied. The Step Time is related to the Scan Rate by:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

Although the program will not let you enter a Step Time shorter than the minimum value, you may still encounter a "Step Time too short" error message. There are two parameter that slow down the potentiostat and thus affect the minimum Step Time. Autoranging of current requires extra time, so a setting of Auto for the Current Range can cause a Step Time too short message. The Acquisition mode in LSV and CV also can affect the speed of the potentiostat. Use AM set to 4/4 for the shortest step times.

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16 Chapter Sixteen Square Wave Voltammetry Setup

Overview

This chapter describes the parameters unique to a Square Wave Voltammetry (SWV) experiment. These parameters define the SWV waveform. For additional setup information, refer to these chapters before or during the SWV setup:

Chapter 6 . . . . . setup menu
Chapter 13 . . . . . common parameters

Square Wave Voltammetry Defined

In this technique, the potentiostat applies a series of forward and reverse pulses sumperimposed on a linear scan of constant height, the Scan Increment (SI). The potential scan begins at the Initial Potential(IP) and ends at the Final Potential (FP). Both the Pulse Height (PH) and the scan increment are constant throughout the experiment. The width of the pulses are inversely related to the Frequency (FR) of the applied pulse sequence, ie. pulse width = 1/2 x 1/FR. The potentiostat samples the current at the end of both the forward and reverse pulses to get the forward and reverse currents. After the experiment you can display the forward current or the reverse current or the difference current or all three. The difference current is very useful for improving sensitivity of analytical measurements.

A typical response curve for the difference current displays a flat baseline with peaks corresponding to the electroactive species. The peak height is directly related to the concentration of these species. The results of this technique are similar to those obtained for Differential Pulse Voltammetry and AC Voltammetry.

Note that, in general, you should not perform experiments with the current range set to **Auto**. It affects the accuracy of the difference current.

Figure 16-1 is a drawing of the applied potential waveform with the experimental parameters labelled:

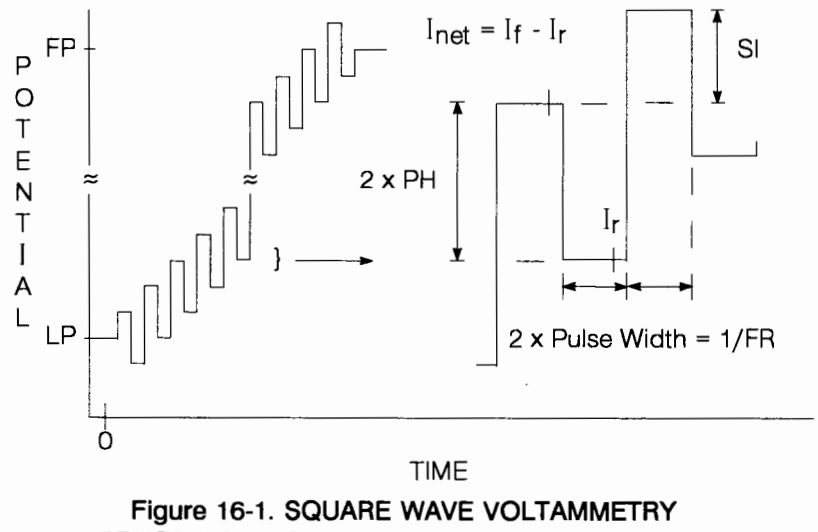


Figure 16-1. SQUARE WAVE VOLTAMMETRY
APPLIED VOLTAGE WAVEFORM AND PARAMETERS

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Final Potential (FP)

Range:

263, 273/273A:	±10.000 V
Versastat, 173 + 276:	±8.000 V

Note: Total scan range must be < 4.0 V.

Default: -1.000 V

Discussion: The Final Potential is the potential at which the scan ends. The value is stored with a resolution of 1 mV and may be entered versus the open circuit potential by adding an 'o' or 'O' character to the end of the value.

The value you enter is corrected to the nearest multiple consistent with the scan increment and initial potential. When you change FP you are also changing the total potential range of the scan which combines with the Scan Increment to determine the Number of Points parameter. Thus, it is possible to get a "Too many points" error message after changing any of your potential limits.

Frequency (FR)

Range:

273/273A	1 mHz to 10 kHz (w/Auto)
263	1 mHz to 4.17 kHz or 500 Hz (w/Auto)
Versastat, 173 + 276	1 mHz to 1.82 kHz or 500 Hz (w/Auto)

Default: 60 Hz

Discussion: The frequency of the square wave is entered in Hz. One cycle consists of a foward pulse and a backward pulse coupled with 2 current measurements. Thus the time per point or step time is equal to the reciprocal of the frequency divided by 2. The allowed values depend on the both the program due to the potentiostat and whether the Current Range is set to Auto. In all cases the lower limit is 0.001 Hz. With autoranging turned off, the upper limit on frequency is 10.0 kHz for the 273/273A, 4.17 kHz for the 263 and 1.82 kHz for the Versastat or 173 + 276. With autoranging turned on (not advised), the upper limit is 500 Hz for all potentiostats.

After changing the Frequency, the program will update its step time setting. If you have entered too high a Frequency, you may get a "Step time too short" error message. Simply decrease the Frequency consistent with the limits cited above.

Note that for an SWV experiment, the scan rate is not a relevant setting, but an effective scan rate can be calculated by multiplying the frequency by the scan increment.

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Initial Potential (IP)

Range:

263, 273/273A:	±10.000 V
Versastat, 173 + 276:	±8.000 V

Default: 0.000 V

Discussion: The Initial Potential is the potential that is applied at the start of the experiment and during Equilibration Time. Values are stored with a resolution of 1mV and the total potential range must be no more than 4 V. Otherwise a "Potentials out of range" error message will result. Also the total potential range during a scanning technique combines with the Scan Increment to determine Number of Points parameter. Thus adjusting the Initial Potential can also lead to a "Too many points" error message. You may enter the value vs. the open circuit potential by simply adding an 'o' or 'O' character at the end of the value.

Number of Points (NP)

Range: 1 to 3000 points

Default: 200

Discussion: The Number of Points (NP) is the total number of points taken during the experiment. This parameter is not user settable for this technique. Its value is shown for informational purposes only. It is controlled by the total potential range of the scan combined with the setting of the Scan Increment.

Pulse Height (PH)

Range: ±2 V

Default: 25 mV V

Discussion: The Pulse in Square Wave Voltammetry is the height of one half-cycle of the square wave pulse excluding the scan increment.

Note that setting a very high Pulse Height can cause a "Potentials out of Range" error message if superimposing the Pulse on the Initial or Final Potential leads to a potential range greater than 4V or an absolute potential greater than 10 V (263, 273/273A) or 8 V (Versastat, 173 + 276).

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Scan Increment (SI)

Range: 1 to 20 mV

Default: 2 mV

Discussion: The Scan Increment determines how much the potential changes between two data points in the experiment. When combined with the potential limits it determines the Number of Points parameter. When combined with the Step Time for the scan techniques it determines the Scan Rate by following equation:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

After changing the Scan Increment, the program will adjust the values of the Number of Points and the Step Time (except for Square Wave where this parameter is irrelevant). Thus, you may encounter either a "Too many points" error message if the Number of Points exceeds 3000 points. The Step Time setting is adjusted based on the current Scan Rate and may generate a "Step time too short" error message.

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17 Chapter Seventeen Sampled DC (SDC) Setup

Overview

This chapter describes the parameters unique to a Sampled DC (SDC) experiment. These parameters define the SDC waveform. For additional setup information, refer to these chapters before or during the SDC setup:

Chapter 6 . . . . . setup menu
Chapter 13 . . . . . common parameters

SDC Defined

In this technique, the potentiostat applies a potential staircase from the initial potential (IP) to the final potential (FP) and samples the current near the end of each step. The height of the steps is the Scan Increment (SI) and the length of each step is the Step/Drop Time (ST). The Scan Rate (SR) is the Scan Increment divided by the Step Time. The software will dispense a new mercury drop at the end of each step if the Working Electrode is set to DME.

Originally this technique was designed for dropping mercury electrodes to avoid recording the large current oscillations due to the growth of the drop between measurements. A typical response curve is sigmoidal indicating the presence of an electroactive species, with the half-wave potential identifying the species and the wave height being related to its concentration.

Figure 17-1 is a drawing of the applied potential waveform with the experimental parameters labelled:

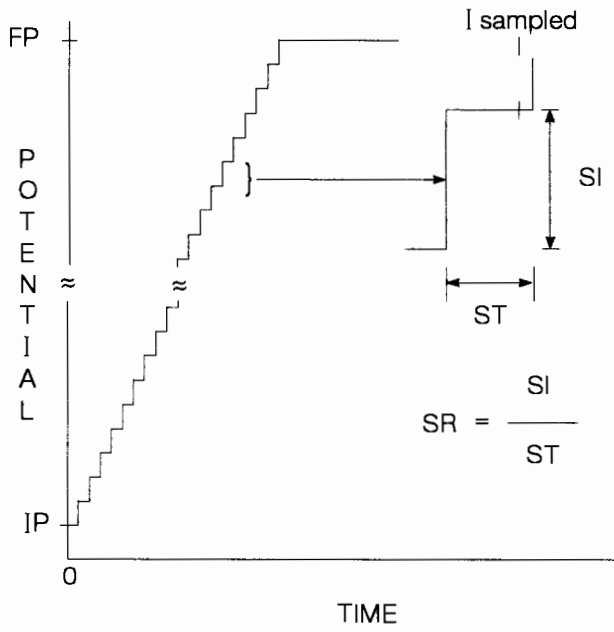


Figure 17-1. SAMPLED DC APPLIED VOLTAGE WAVEFORM AND PARAMETERS

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Final Potential (FP)

Range:

263, 273/273A	±10.000 V
Versastat, 173 + 276	±8.000 V

Note: Total scan range must be < 4.0 V.

Default: -1.000 V

Discussion: The Final Potential is the potential at which the scan ends. The value is stored with a resolution of 1 mV and may be entered versus the open circuit potential by adding an 'o' or 'O' character to the end of the value.

The value you enter is corrected to the nearest multiple consistent with the scan increment and initial potential. When you change FP you are also changing the total potential range of the scan which combines with the Scan Increment to determine the Number of Points parameter. Thus, it is possible to get a "Too many points" error message after changing any of your potential limits.

Initial Potential (IP)

Range:

263, 273/273A:	±10.000 V
Versastat, 173 + 276:	±8.000 V

Default: 0.000 V

Discussion: The Initial Potential is the potential that is applied at the start of the experiment and during the Pre-Electrolysis Time (CPC) or Equilibration Time (all other techniques). Values are stored with a resolution of 1mV and the total potential range must be no more than 4 V. Otherwise a "Potentials out of range" error message will result. Also the total potential range during a scanning technique combines with the Scan Increment to determine Number of Points parameter. Thus adjusting the Initial Potential can also lead to a "Too many points" error message. You may enter the value vs. the open circuit potential by simply adding an 'o' or 'O' character at the end of the value.

Scan Increment (SI)

Range: 1 to 20 mV
Default: 2 mV

Discussion: The Scan Increment determines how much the potential changes between two data points in the experiment. When combined with the potential limits it determines the Number of Points parameter. When combined with the Step Time for the scan techniques it determines the Scan Rate by following equation:

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$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

After changing the Scan Increment, the program will adjust the values of the Number of Points and the Step Time (except for Square Wave where this parameter is irrelevant). Thus, you may encounter either a "Too many points" error message if the Number of Points exceeds 3000. The Step Time setting is adjusted based on the current Scan Rate and may generate a "Step time too short" error message.

Scan Rate (SR)

P'stat:	Model 273/273A	Model 263	Versastat and Model 173 + Model 276
Minimum	1E-6 V/s	1E-6 V/s	1E-6 V/s
Maximum			
Calculated	400 V/s	166.7 V/s	72 V/s
Recommended	160 V/s	66.7 V/s	29 V/s
Default:	20E-3 V/s	20E-3 V/s	20E-3 V/s

Discussion: The scan rate controls how quickly the potential changes in the voltammetric techniques. It is directly related to the scan increment and the step time:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

After the scan rate is changed, the program updates the step time using the values for scan rate and scan increment.

Step/Drop Time (ST)

Range:

273/273A:	50 μs to 1,074 s
263:	120 μs to 1074 s
Versastat, 173 + 276:	275 μs to 1,074 s

Default: 0.1 s

Discussion: The Step Time setting determines how long each Scan Increment is applied. In addition for the polarography techniques (SDC, NPP, RNP and DPV with a DME working electrode), it determines the lifetime of a Hg drop. For the polarographic techniques lifetime of the drop may be erratic if the Step/Drop Time is set to less than 0.1 s due to limitations imposed by rate of communication between the computer and the potentiostat limitation due to the mechanical properties of the Model 303A. The Step Time is related to the Scan Rate by:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

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Although the program will not let you enter a Step Time shorter than the minimum value, you may still encounter a "Step Time too short" error message. There are two parameter that slow down the potentiostat and thus affect the minimum Step Time. Autoranging of current requires extra time, so a setting of Auto for the Current Range can cause a Step Time too short message.

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18 Chapter Eighteen Differential Pulse (DP) Setup

Overview

This chapter describes the parameters unique to a Differential Pulse (DP) experiment. These parameters define the DP waveform. For additional setup information, refer to these chapters before or during the DP setup:

Chapter 6 . . . . . setup menu
Chapter 13 . . . . . common parameters

Differential Pulse Defined

In this technique, the applied waveform is the sum of a pulse train and a staircase from the Initial Potential (IP) to the Final Potential (FP). The pulses have a width, Pulse Width (PW), and a height, Pulse Height (PH). The height of the step in the staircase is the Scan Increment (SI) and the widht of the steps is the Step Time (ST).

The potentiostat samples the current just before a pulse (Istep) and near the end of the pulse (Ipulse). A typical response curve plots the difference in these two currents and has a relatively flat baseline with peaks corresponding to the electroactive species. The curve shape is very similar to that produced in Square Wave and AC Voltammetry. This technique is more useful for analytical determinations than Normal Pulse or Sampled DC, since its differential measurements discriminates against charging currents leading to enhanced sensitivity. The peak height is directly related to the concentration of the electroactive species.

Figure 18-1 is a drawing of the applied potential waveform with the experimental parameters labelled:

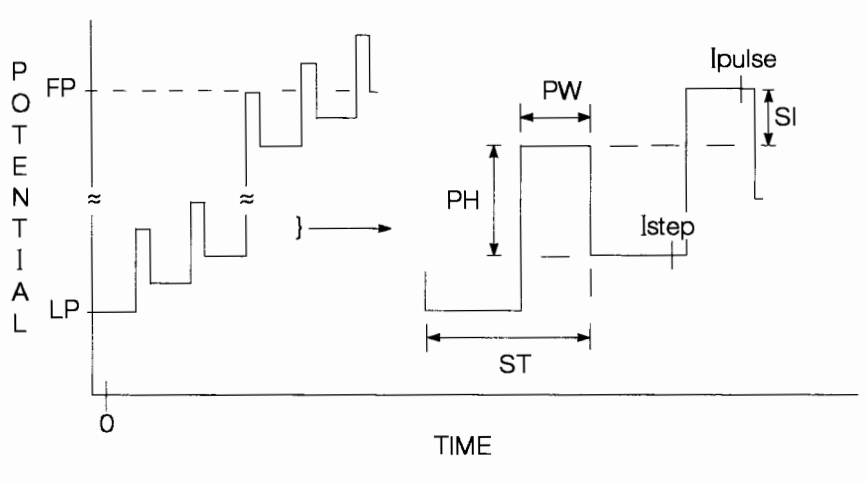


Figure 18-1. DIFFERENTIAL PULSE
APPLIED VOLTAGE WAVEFORM AND PARAMETERS

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Final Potential (FP)

Range:

263, 273/273A	±10.000 V
Versastat, 173 + 276	±8.000 V

Note: Total scan range must be < 4.0 V.

Default: -1.000 V

Discussion: The Final Potential is the potential at which the scan ends. The value is stored with a resolution of 1 mV and may be entered versus the open circuit potential by adding an 'o' or 'O' character to the end of the value.

The value you enter is corrected to the nearest multiple consistent with the scan increment and initial potential. When you change FP you are also changing the total potential range of the scan which combines with the Scan Increment to determine the Number of Points parameter. Thus, it is possible to get a "Too many points" error message after changing any of your potential limits.

Initial Potential (IP)

Range:

263, 273/273A:	±10.000 V
Versastat, 173 + 276:	±8.000 V

Default: 0.000 V

Discussion: The Initial Potential is the potential that is applied at the start of the experiment and during the Pre-Electrolysis Time (CPC) or Equilibration Time (all other techniques). Values are stored with a resolution of 1mV and the total potential range must be no more than 4 V. Otherwise a "Potentials out of range" error message will result. Also the total potential range during a scanning technique combines with the Scan Increment to determine Number of Points parameter. Thus adjusting the Initial Potential can also lead to a "Too many points" error message. You may enter the value vs. the open circuit potential by simply adding an 'o' or 'O' character at the end of the value.

Pulse Height (PH)

Range: ±2 V

Default: 25 mV

Discussion: The Pulse Height in differential pulse it equal to the height of pulse applied just before the end of the step time at the start of the pulse width.

Note that setting a very high Pulse Height can cause a "Potentials out of Range" error message if superimposing the Pulse on the Initial or Final Potential leads to

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a potential range greater than 4V or an absolute potential greater than 10 V (263, 273/273A) or 8 V (Versastat, 173 + 276).

Pulse Width (PW)

Range:

273/273A	50 μs to 400 s
263	120 μs to 400 s
Versastat, 173 + 276	275 μs to 400 s

Default: 50 ms

Discussion: The pulse width is the length of the pulse that occurs just before the end of the step/drop time. The current is sampled just before and at the end of the pulse. For this reason, the Step/Drop Time is always at least twice the length of the Pulse Width and the program will adjust the Step/Drop Time as needed.

Note that if the Pulse Width is changed the program will also adjust the scan rate and number of points to make them consistent with the changes made to Pulse Width and the Step Time.

Scan Increment (SI)

Range: 1 to 20 mV

Default: 2 mV

Discussion: The Scan Increment determines how much the potential changes between two data points in the experiment. When combined with the potential limits it determines the Number of Points parameter. When combined with the Step Time for the scan techniques it determines the Scan Rate by following equation:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

After changing the Scan Increment, the program will adjust the values of the Number of Points and the Step Time. Thus, you may encounter either a "Too many points" error message if the Number of Points exceeds 3000 points. The Step Time setting is adjusted based on the current Scan Rate and may generate a "Step time too short" error message.

Scan Rate (SR)

P'stat:	Model 273/273A	Model 263	Versastat and Model 173 + Model 276
Minimum	1E-6 V/s	1E-6 V/s	1E-6 V/s
Maximum			
Calculated	400 V/s	166.7 V/s	72 V/s
Recommended	160 V/s	66.7 V/s	29 V/s

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Default: 20E-3 V/s 20E-3 V/s 20E-3 V/s

Discussion: The scan rate controls how quickly the potential changes in the voltammetric techniques. It is directly related to the scan increment and the step time:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

After the scan rate is changed, the program updates the step time using the values for scan rate and scan increment.

Step/Drop Time (ST)

Range:

273/273A:	50 μs to 1074 s
263:	120 μs to 1074 s
Versastat, 173 + 276:	275 μs to 1074 s

Default: 0.1 s

Discussion: The Step Time setting determines how long each Scan Increment is applied. In addition for the polarography techniques (SDC, NPP, RNP and DPV with a DME working electrode), it determines the lifetime of a Hg drop. For the polarographic techniques lifetime of the drop may be erratic if the Step/Drop Time is set to less than 0.1 s due to limitations imposed by rate of communication between the computer and the potentiostat limitation due to the mechanical properties of the Model 303A. The Step Time is related to the Scan Rate by:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

Although the program will not let you enter a Step Time shorter than the minimum value, you may still encounter a "Step Time too short" error message. There are two parameter that slow down the potentiostat and thus affect the minimum Step Time. Autoranging of current requires extra time, so a setting of Auto for the Current Range can cause a Step Time too short message.

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19 Chapter Nineteen Normal Pulse (NP) Setup

Overview

This chapter describes the parameters unique to a Normal Pulse (NP) experiment. These parameters define the NP waveform. For additional setup information, refer to these chapters before or during the NP setup:

Chapter 6 . . . . . setup menu
Chapter 13 . . . . . common parameters

Normal Pulse Defined

In this technique, the potentiostat applies a series of potential pulses from a constant baseline equal to the Initial Potential (IP). As the experiment progresses the height of the pulses ranges from 0 to the difference between IP and the Final Potential (FP). The total cycle time between pulses is the Step Time (ST) and is independent of the Pulse Width (PW). The potentiostat samples the current just before each new pulse and near the end of the pulse and measures the difference between the two values. The amount the pulse height increases with each new step is the Scan Increment (SI).

A typical response curve is sigmoidal with waves representing the presence of electroactive species while the wave height is directly proportional to the concentration of the electroactive species. It was orignally intended to enhance the current response over Sampled DC by avoiding continual depletion of the double layer and the solution in the vicinity of the electrode. Although it gives a more sensitive response than Sampled DC, noise tends be a problem at low concentrations limiting its sensitivity.

Figure 19-1 is a drawing of the applied potential waveform with the experimental parameters labelled:

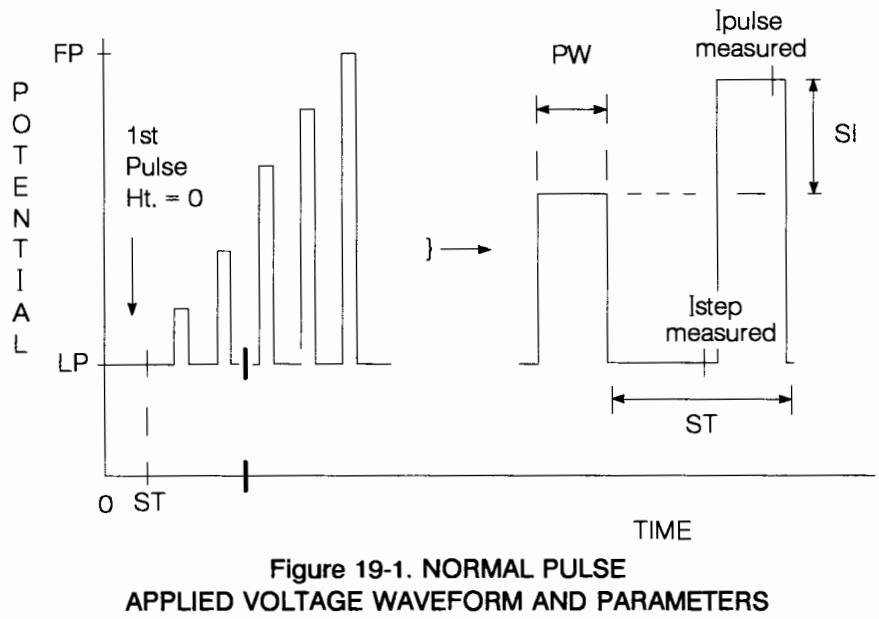


Figure 19-1. NORMAL PULSE APPLIED VOLTAGE WAVEFORM AND PARAMETERS

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Final Potential (FP)

Range:

263, 273/273A:	±10.000 V
Versastat, 173 + 276:	±8.000 V

Note: Total scan range must be < 4.0 V.

Default: -1.000 V

Discussion: The Final Potential is the potential at which the scan ends. The value is stored with a resolution of 1 mV and may be entered versus the open circuit potential by adding an 'o' or 'O' character to the end of the value.

The value you enter is corrected to the nearest multiple consistent with the scan increment and initial potential. When you change FP you are also changing the total potential range of the scan which combines with the Scan Increment to determine the Number of Points parameter. Thus, it is possible to get a "Too many points" error message after changing any of your potential limits.

Initial Potential (IP)

Range:

263, 273/273A:	±10.000 V
Versastat, 173 + 276:	±8.000 V

Default: 0.000 V

Discussion: The Initial Potential is the potential that is applied at the start of the experiment and during the Equilibration Time. Values are stored with a resolution of 1mV and the total potential range must be no more than 4 V. Otherwise a "Potentials out of range" error message will result. Also the total potential range during a scanning technique combines with the Scan Increment to determine Number of Points parameter. Thus adjusting the Initial Potential can also lead to a "Too many points" error message. You may enter the value vs. the open circuit potential by simply adding an 'o' or 'O' character at the end of the value.

Pulse Width (PW)

Range:

273/273A	50 μs to 400 s
263	120 μs to 400 s
Versastat, 173 + 276	275 μs to 400 s

Default: 50E-3 s

Discussion: The pulse width is the length of the pulse that occurs just before the end of the step/drop time. The current is sampled just before and at the end of the pulse. For this reason, the Step/Drop Time is always at least twice the length of the

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Pulse Width and the program will adjust the Step/Drop Time as needed. The pulse height varies between 0 and the difference between the initial and final potentials.

Note that if the Pulse Width is changed the program will also adjust the scan rate and number of points to make them consistent with the changes made to Pulse Width and the Step Time.

Scan Increment (SI)

Range: 1 to 20 mV

Default: 2 mV

Discussion: The Scan Increment determines how much the potential changes between two data points in the experiment. When combined with the potential limits it determines the Number of Points parameter. When combined with the Step Time for the scan techniques it determines the Scan Rate by following equation:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

After changing the Scan Increment, the program will adjust the values of the Number of Points and the Step Time (except for Square Wave where this parameter is irrelevant). Thus, you may encounter a "Too many points" error message if the Number of Points exceeds 3000 points. The Step Time setting is adjusted based on the current Scan Rate and may generate a "Step time too short" error message.

Scan Rate (SR)

P'stat:	Model 273/273A	Model 263	Versastat and Model 173 + Model 276
Minimum	1E-6 V/s	1E-6 V/s	1E-6 V/s
Maximum			
Calculated	400 V/s	166.7 V/s	72 V/s
Recommended	160 V/s	66.7 V/s	29 V/s
Default:	20E-3 V/s	20E-3 V/s	20E-3 V/s

Discussion: The scan rate controls how quickly the potential changes in the voltammetric techniques. It is directly related to the scan increment and the step time:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

After the scan rate is changed, the program updates the step time using the values for scan rate and scan increment.

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Step/Drop Time (ST)

Range:

273/273A:	50 μs to 1074 s
263:	120 μs to 1074 s
Versastat, 173 + 276:	275 μs to 1074 s

Default: 0.1 s

Discussion: The Step Time setting determines how long each Scan Increment is applied. In addition for the polarography techniques (SDC, NPP, RNP and DPV with a DME working electrode), it determines the lifetime of a Hg drop. For the polarographic techniques lifetime of the drop may be erratic if the Step/Drop Time is set to less than 0.1 s due to limitations imposed by rate of communication between the computer and the potentiostat limitation due to the mechanical properties of the Model 303A. The Step Time is related to the Scan Rate by:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

Although the program will not let you enter a Step Time shorter than the minimum value, you may still encounter a "Step Time too short" error message. There are two parameter that slow down the potentiostat and thus affect the minimum Step Time. Autoranging of current requires extra time, so a setting of Auto for the Current Range can cause a Step Time too short message.

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20 Chapter Twenty Reverse Normal Pulse Setup

Overview

This chapter describes the parameters unique to a Reverse Normal Pulse (RNP) experiment. These parameters define the RNP waveform. For additional setup information, refer to these chapters before or during the RNP setup:

Chapter 6 . . . . . setup menu
Chapter 13 . . . . . common parameters

Reverse Normal Pulse Defined

In this technique, the potentiostat applies a series of potential pulses of constant baseline but increasing amplitude. However, unlike the Normal Pulse technique, the baseline potential is the final potential and the pulse steps are toward the initial potential start small and building toward the difference in the initial and final potential. The response curve plots a difference current and is again sigmoidal as in Normal Pulse, but the curve can have both anodic and cathodic limiting plateaus depending on reversibility of the couple.

This technique is very similar to the Normal Pulse technique in that the potentiostat applies a series of potential pulses of varying height from a constant baseline corresponding to the Initial Potential (IP). For both techniques, the height of the pulses ranges from 0 to the difference between IP and the Final Potential (FP) as the experiment progresses.

The difference between the two techniques is mostly in what is considered the direction of the "pulse" and in the amount of time spent at each potential. The total cycle time between pulses is the Step Time (ST) and is independent of the Pulse Width (PW). In Normal Pulse the potential is stepped from IP toward FP for a length of time corresponding to the pulse width with the potential being at IP for the rest of the step time. Thus the pulse is viewed as being from IP toward FP. In Reverse Normal Pulse, an almost identical waveform is used but the pulse potential of Normal Pulse is now considered a step potential and the pulse is viewed as being from this step potential toward IP. In RNP the majority of the step time is spent at the step potential and only returned to IP for a short time PW. The amount the pulse height and step potential increase with each new step is the Scan Increment (SI).

This difference in the two techniques pulse definition affects the net or difference current. In Normal Pulse, the net current is the current at the end of the pulse minus any residual current at IP at the end of the previous potential step. In the case of Reverse Normal Pulse, this is also true, but the numbers get rearranged due to the differing outlook on what is the pulse. In RNP, the pulse current is viewed as occurring at IP and its step current is what Normal Pulse considered the pulse current. Thus plots of the difference current, $I_{pulse} - I_{step}$, has the opposite sign when using similar setups in both RNP and Normal Pulse experiments. A typical response

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curve in RNP is reverse sigmoidal with waves representing the presence of electroactive species. One of the advantages of RNP is that it is more sensitive to slow kinetic complications than Normal Pulse.

Figure 20-1 below is a drawing of the applied potential waveform with the experimental parameters labelled:

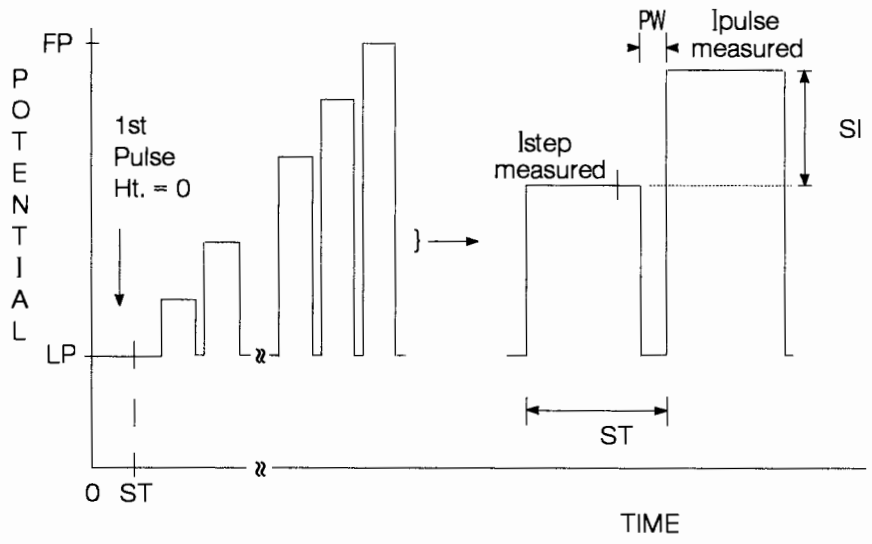


Figure 20-1. REVERSE NORMAL PULSE APPLIED VOLTAGE WAVEFORM AND PARAMETERS

Final Potential (FP)

Range:
263, 273/273A: ±10.000 V
Versastat, 173 + 276: ±8.000 V
Note: Total scan range must be < 4.0 V.

Default: -1.000 V

Discussion: The Final Potential is the potential at which the scan ends. The value is stored with a resolution of 1 mV and may be entered versus the open circuit potential by adding an 'o' or 'O' character to the end of the value.

The value you enter is corrected to the nearest multiple consistent with the scan increment and initial potential. When you change FP you are also changing the total potential range of the scan which combines with the Scan Increment to determine the Number of Points parameter. Thus, it is possible to get a "Too many points" error message after changing any of your potential limits.

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Initial Potential (IP)

Range:

263, 273/273A:	±10.000 V
Versastat, 173 + 276:	±8.000 V

Default: 0.000 V

Discussion: The Initial Potential is the potential that is applied at the start of the experiment and during the Pre-Electrolysis Time (CPC) or Equilibration Time (all other techniques). Values are stored with a resolution of 1mV and the total potential range must be no more than 4 V. Otherwise a "Potentials out of range" error message will result. Also the total potential range during a scanning technique combines with the Scan Increment to determine Number of Points parameter. Thus adjusting the Initial Potential can also lead to a "Too many points" error message. You may enter the value vs. the open circuit potential by simply adding an 'o' or 'O' character at the end of the value.

Pulse Width (PW)

Range:

273/273A	50 μs to 400 s
263	120 μs to 400 s
Versastat, 173 + 276	275 μs to 400 s

Default: 50 ms

Discussion: The pulse width is the length of the pulse that occurs just before the end of the step/drop time. The current is sampled just before and at the end of the pulse. For this reason, the Step/Drop Time is always at least twice the length of the Pulse Width and the program will adjust the Step/Drop Time as needed. The pulse height varies between 0 and the difference between the initial and final potentials.

Note that if the Pulse Width is changed the program will also adjust the scan rate and number of points to make them consistent with the changes made to Pulse Width and the Step Time.

Scan Increment (SI)

Range: 1 to 20 mV

Default: 2 mV

Discussion: The Scan Increment determines how much the potential changes between two data points in the experiment. When combined with the potential limits it determines the Number of Points parameter. When combined with the Step Time for the scan techniques it determines the Scan Rate by following equation:

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$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

After changing the Scan Increment, the program will adjust the values of the Number of Points and the Step Time (except for Square Wave where this parameter is irrelevant). Thus, you may encounter either a "Too many points" error message if the Number of Points exceeds 3000 points. The Step Time setting is adjusted based on the current Scan Rate and may generate a "Step time too short" error message.

Scan Rate (SR)

P'stat:	Model 273/273A	Model 263	Versastat and Model 173 + Model 276
Minimum	1E-6 V/s	1E-6 V/s	1E-6 V/s
Maximum			
Calculated	400 V/s	166.7 V/s	72 V/s
Recommended	160 V/s	66.7 V/s	29 V/s
Default:	20E-3 V/s	20E-3 V/s	20E-3 V/s

Discussion: The scan rate controls how quickly the potential changes in the voltammetric techniques. It is directly related to the scan increment and the step time:

$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

After the scan rate is changed, the program updates the step time using the values for scan rate and scan increment.

Step/Drop Time (ST)

Range:

273/273A:	50 μs to 1074 s
263:	120 μs to 1074 s
Versastat, 173 + 276:	275 μs to 1074 s

Default: 0.1 s

Discussion: The Step Time setting determines how long each Scan Increment is applied. In addition for the polarography techniques (SDC, NPP, RNP and DPV with a DME working electrode), it determines the lifetime of a Hg drop. For the polarographic techniques lifetime of the drop may be erratic if the Step/Drop Time is set to less than 0.1 s due to limitations imposed by rate of communication between the computer and the potentiostat limitation due to the mechanical properties of the Model 303A. The Step Time is related to the Scan Rate by:

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$$Scan\ Rate = \frac{Scan\ Increment}{Step\ Time}$$

Although the program will not let you enter a Step Time shorter than the minimum value, you may still encounter a "Step Time too short" error message. There are two parameter that slow down the potentiostat and thus affect the minimum Step Time. Autoranging of current requires extra time, so a setting of Auto for the Current Range can cause a Step Time too short message.

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21 Chapter Twenty-One Chronoamperometry (CA) & Chronocoulometry (CC)

Overview

This chapter describes the parameters unique to Chronoamperometry (CA) and Chronocoulometry (CC) experiments. These parameters define the CA and CC waveforms. For additional setup information, refer to these chapters before or during a CA or CC setup:

Chapter 6 . . . . . setup menu
Chapter 13 . . . . . common parameters

Chronoamperometry (CA) and Chronocoulometry (CC): Defined and Compared

Chronoamperometry (CA) and Chronocoulometry (CC) are very similar experiments that use the same applied potential waveform shown in Figure 21-1. In both the potentiostat steps the potential from an Initial Potential (IP) to Potential Step 1 (E1) for a length of time, Time Step 1 (T1). Double potential step experiments may also be performed by setting the Potential Step 2 (E2) and Time Step 2 (T2) to values other than PASS.

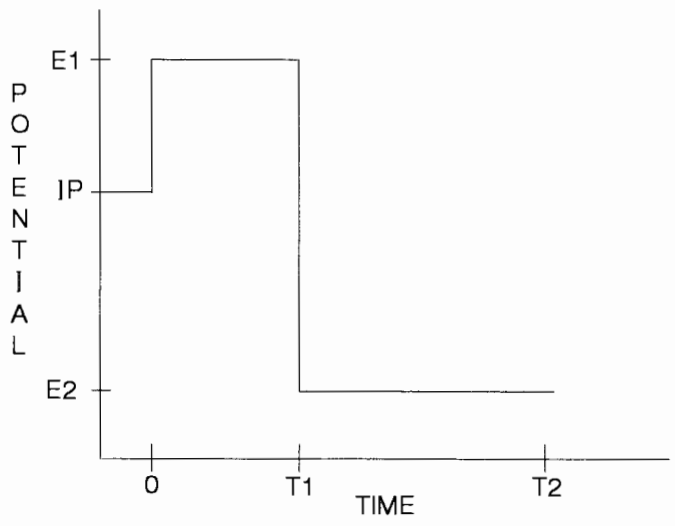


Figure 21-1. CHRONOAMPEROMETRY APPLIED VOLTAGE WAVEFORM AND PARAMETERS

The key difference in the experiment performed is in the type of data collected. CA records current observed as a function of time, while CC records integrated current or charge as a function of time. Both are useful techniques for investigating

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chemical kinetics as well as measuring diffusion coefficients. In the case of CA a typical response curve displays a sharp current spike which decays with time. The typical response curve for CC is just the integral of the CA response curve and usually rises rapidly from approximately 0 charge to approach a limiting value as time increases. The similarity of the experiments is enhanced by the availability of a calculated integral style for CA (Q vs. t displayed from i vs. t data) and a calculated derivative style for CC (i vs. t displayed from Q vs. t data).

If these experiments are so similar, are there any real differences? Yes, on a hardware level they are very different. The CC technique makes use of a hardware integrator and actually samples Q as collected on a capacitor. This hardware and therefore this technique is only available when your potentiostat is a Model 273A or a Model 273 equipped with a /96 option board. If you are using this equipment, this technique is available. With other potentiostats or on a 273 potentiostat not equipped with the /96 option board, Model 270 software will refuse to run a CC experiment, since the potentiostat would not be able to accept the commands needed to carry it out. Chronoamperometry, on the other hand, runs on all potentiostats as it only samples the current that flows during the experiment, not the charge.

This difference in how the experiment is performed on a hardware level has an effect on both maximum charge that can be measured and on the timescales available. During a CC experiment, the maximum charge that can be measured is determined by the MAX Q parameter and is analogous to the Current Range setting but applies to the charge observed. The choices for this parameter are based on the hardware limitations of the /96 option board as well as the present Current Range setting. If the charge measured in a given experiment exceeds MAX Q, a charge overload will be observed as an abrupt flattening of the response curve. Note that current overloads are still possible in chronocoulometry and are not discernible in the CC Q vs. t plot style, but the data is still invalid. For this reason, you should get in the habit of viewing the i vs. t trace of CC data to check for current overloads. Charge overloads are not a problem when current data is collected in a chronoamperometry experiment.

These two techniques also differ in the practical timescale settings available. The average current that flows during your experiment will dictate the appropriate current range for the experiment and will also the determine the largest setting for the MAX Q parameter. This maximum charge value will limit the amount of time this current can flow. For this reason, chronocoulometry is more useful for short timescale experiments (microseconds to seconds). Chronoamperometry, on the other hand, has no such limitation on the timescale of the experiment and the total length of the experiment can range from microseconds to months. Thus for long timescale experiments involving large amounts of charge and current being passed, your only choice is to run CA and then redisplay the data as Q vs. t. For short timescales, though, you can choose between CA and CC.

For short timescales, does it really matter which technique is used? Yes. Why? If you run a CA experiment, the program can only calculate the charge based on the average current that was flowing at point the current was sampled. It cannot show you the true instaneous charge, since this calculation is different from an actual hardware integration of the current that flowed. Likewise, when you run a CC experiment, the program can only calculate the average current for each data interval and display it. It cannot not display the actual instantaneous current.

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Okay, that makes some sense, but it still seems like this is actually a fine point and this difference between CA and CC does not really matter. Wrong! Redisplaying the data as calculated i or Q may not give you an accurate representation of the calculated quantity even though the math is performed correctly. For example, a large current spike occurs in both techniques when the potential is changed. This current spike is properly integrated in chronocoulometry due to the hardware circuitry on the /96 board. In chronoamperometry, the current is only sampled not integrated on the hardware level. The true current maximum will most likely fall between actual data points. Depending on the amount of charge observed in this first short jump of the current, the actual charge measured in hardware integration will be very different from that calculated by software integration of the sampled current data. As a result the Q vs. t plot of a CA experiment will differ from the Q vs. t curve actually recorded in a CC experiment. In the case of a study of adsorption by chronocoulometry where the charge passed at t=0 represents charge due to surface phenomena, this discrepancy would greatly affect any conclusions you might try to draw based on CA data displayed as Q vs. t. If you were to compare CA data as displayed as Q vs. t with actual CC data displayed as Q vs. t, you would find a smaller Q at t=0 due to the lack of hardware integration.

Often the only reason pulse experiments are performed is to measure the rate of chemical reactions. The papers published usually predict instaneous charge or current values not the average values of these quanitities at any point in time. If you have a paper that gives numbers for the effect of kinetics on chronoamperometry or chronocoulometry, you should run the exact experiment the paper analyzes, so that you are collecting instaneous values. If you do wish to try to analyze the calculated integral or derivative response curve, then you should try to either minimize the Time Per Point parameter or maximize the Number of Points for a given experimental timescale. Better resolution on the sampled data will help minimize any discrepancies between between calculated values (i or Q) and the true unmeasured quantities (Q or i). In addition these discrepancies are not as important at longer timescales.

To summarize, when performing pulse experiments to extract kinetic information, you should perform the exact experiment that you wish to analyze. You should not perform CA and analyze the software integrated Q vs. t curve or perform CC and analyze the software derivative i vs. t curve. If you decide to analyze the data anyway, you are advised to maximize the data resolution to minimize any discrepancies that could arise between calculated values and the true, unmeasured values.

Initial Potential (IP)

Range:

263, 273/273A:	±10.000 V
Versastat, 173 + 276:	±8.000 V

Default: 0.000 V

Discussion: The Initial Potential is the potential that is applied at the start of the experiment and during the Equilibration Time. Values are stored with a resolution

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of 1mV and the total potential range must be no more than 4 V. Otherwise a "Potentials out of range" error message will result. Also the total potential range during a scanning technique combines with the Scan Increment to determine Number of Points parameter. Thus adjusting the Initial Potential can also lead to a "Too many points" error message. You may enter the value vs. the open circuit potential by simply adding an 'o' or 'O' character at the end of the value.

For CA and CC experiments the Potential Step 2 (E2) parameter can be locked to IP. What that means is that if IP is the same as E2 and you change IP, then E2 is updated to the same value. This is done to reflect that in these experiments it is very common to have the last potential applied to the cell be the same as the initial potential. You can unlock E2 by simply changing its value to something different from IP.

MAX Q (Maximum Charge, CC only)

Range: 80 fC to 400 mC (depends on current range)

Default: 400 uC

Discussion: The MAX Q setting is a Q range setting specific to the charge passed in the chronocoulometry experiment in much the same way that the current range is related to the maximum current passed in all experiments. The choices for MAX Q are tied to the presently selected Current Range, so your options vary with the Current Range setting. Note that if you make a selection here and then change the current range, the program may be forced to change MAX Q to a new value consistent with the new current range.

If the charge exceeds the MAXQ setting exceeded during the experiment, you will observe a "charge overload", although neither the program or the potentiostat will report any errors. In that case, increase MAXQ and rerun the experiment. Note also that current overloads do not necessarily lead to charge overloads, but the data is still invalid. You should get in the habit of always viewing the I vs t plot for chronocoulometry experiments to check for current overloads as they are not discernible in the Q vs t plot. If you have a current overload, increase the Current Range setting and rerun the experiment. Finally, if your Q vs t looks like stair steps, you need to decrease MAXQ to get better data resolution.

Number of Points (NP)

Range: 1 to 3000 points

Default: 200

Discussion: The Number of Points(NP) is the total number of points taken during the experiment. For most of the experimental techniques, this is not user settable, but can be entered for the time based techniques, chronoamperometry, chronocoulometry, chronopotentiometry and controlled potential coulometry. For these experiments NP includes 10 data points collected at the initial conditions before the first potential or current step.

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Note after you change NP, the program adjusts the Time per Point (TP) by dividing the total experiment time by the newly entered number of points less 10 points and correcting TP to the nearest multiple consistent allowed potentiostat values for the TP parameter.

Potential Step 1 & 2 (E1 & E2)

Range:

263, 273/273A:	±10.000 V, Pass (E2 only)
Versastat, 173 + 276:	±8.000 V, Pass (E2 only)

Default: 0.100 V

Discussion: The E1 and E2 settings control the size of the first and second potential step in chronoamperometry and chronocoulometry. Enter a value in volts in multiples of .001 V or enter 'p' to set to PASS.

As for all potential settings in any experiment, the whole potential range must be under 4 V. If you enter values with more than a 4 V range, you will get a "Potentials out of range" error message. The program will also refuse to the experiment.

Note that E2 can be is locked to IP by setting them equal to the same value. After that if you change IP, E2 is updated with the same value. To unlock the two values, simply change E2 to desired value.

Note also that E2 & T2 are coupled. If T2 is set to 0 or PASS, E2 is changed to PASS. Furthermore, if T2 is changed to a value other than 0 and E2 is set to PASS, then E2 is set equal to IP, the initial potential. Also if E2 is set to Pass, then T2 is updated to Pass and if E2 is changed from pass then T2 is set equal to T1.

Time per Point (TP)

Range:

273/273A	50 μs to 1074 s or T1
263	120 μs to 1074 s or T1
Versastat, 173 + 276	275 μs to 1074 s or T1

Default: 0.1 s

Discussion: The Time per Point (TP) is just the period of time between the sampling of two data points. It cannot exceed the value of the Time of Step 1 (T1) and the program enforces that limitation. Enter the value in seconds (s) in multiples of 1E-6.

After changing TP, the Number of Points is updated based on the total time of the experiment and the new setting. Note that the Number of Points always includes 10 data points collected at the initial conditions before the first potential step.

Also note that if the Number of Points would exceed the maximum allowed, the

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program adjusts NP to the maximum and sets TP to the minimum value possible based on the total time of the experiment. The program may also adjust TP to make it consistent with potentiostat settings as well.

Time Step 1 & 2 (T1 & T2)

Range:

T1:	TP to 3.221E6 s
T2:	0 to (3.221E6 - T1)

Default: 10 s (T1) PASS (T2)

Discussion: T1 & T2 correspond to the duration of the first and second step of the waveform. Since there would be no first step if T1 were 0, the minimum value you can assign to it is equal to the Time per Point (TP). Enter a value in seconds in multiples of 1E-4 s.

After T1 or T2 changes the program updates the Time per Point based on the total time of the experiment and the Number of Points less 10 data points, since the Number of Points always includes 10 data points collected at the initial conditions before the first potential or current step. If the new value of TP is less than its minimum allowed value, a "Step time too short" error message will occur.

Note that T2 can be is locked to T1 by setting them equal to the same value. After that if you change T1, T2 is updated with the same value. To unlock the two values, simply change T2 to value desired.

Note also that E2 & T2 are coupled. If T2 is set to 0 or PASS, E2 is changed to PASS. Furthermore, if T2 is changed to a value other than 0 and E2 is set to PASS, then E2 is set equal to IP, the initial potential. Also if E2 is set to Pass, then T2 is updated to Pass and if E2 is changed from pass then T2 is set equal to T1.

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22 Chapter Twenty-Two Chronopotentiometry

Overview

This chapter describes the parameters unique to a Chronopotentiometry (CP) experiment. These parameters define the CP waveform. For additional setup information, refer to these chapters before or during the CP setup:

Chapter 6 . . . . . setup menu
Chapter 13 . . . . . common parameters

Chronopotentiometry (CP) Defined

In chronopotentiometry, the potentiostat applies a constant current(I1) for a specified time (T1) and monitors the resulting potential. It can be used for many different types of analyses or to investigate electrode kinetics. It is generally considered less sensitive than voltammetric techniques for analytical uses. The response curve is a sigmoidal plot of potential versus time with various plateaus. Each plateau corresponds to the redox potential of an electroactive species. The time take to traverse the plateau is known as the transition time and can be related to the concentration and diffusion coefficient of the electroactive species.

Figure 22-9 shows the applied current waveform with the experimental parameters labelled:

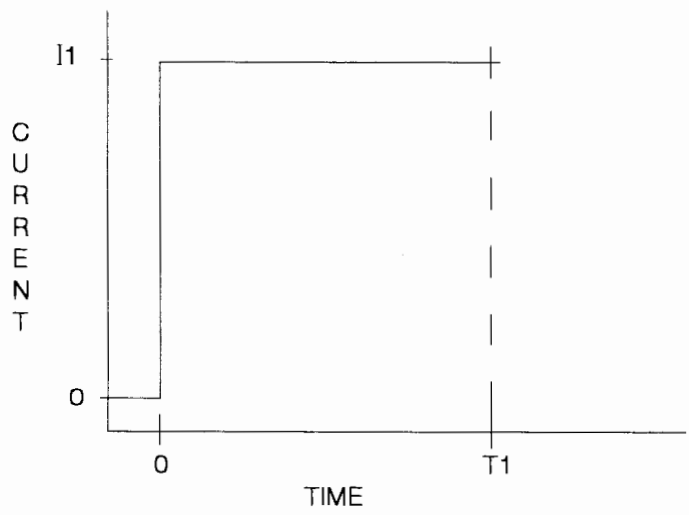


Figure 22-1. CHRONOPOTENTIOMETRY APPLIED CURRENT WAVEFORM WITH PARAMETERS

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Current Step (I1)

Range:

263, 273/273A:	±21E-9 A to ±1 A
Versastat, 173 + 276:	± 1E-6 A to ±1 A

Default: 0.100 A

Discussion: The Current Step 1(I1) setting determines size of the current step in chronopotentiometry. Its limits are program dependent due to potentiostat limitations. Note that although you can enter a value of 0 amperes in the program, the program will not actually use it. Instead it will set I1 to the minimum positive current step.

Maximum Potential (ME)

Range: 0 to 8 V

Default: +8 V

Discussion: The Maximum Potential (ME) setting keeps the chronopotentiometry technique from exposing your working electrode to excessive potentials. If the absolute value of the potential measured during the experiment exceeds the ME setting the experiment is terminated. Note that this feature is controlled by the software not the potentiostat. Consequently the termination of the experiment is not instantaneous and may take a few seconds.

Number of Points (NP)

Range: 1 to 3000 points

Default: 200

Discussion: The Number of Points(NP) is the total number of points taken during the experiment. For most of the experimental techniques, this is not user settable, but can be entered for the time based techniques, chronoamperometry, chronocoulometry, chronopotentiometry and controlled potential coulometry. For these experiments NP includes 10 data points collected at the initial conditions before the first potential or current step.

Note after you change NP, the program adjusts the Time per Point (TP) by dividing the total experiment time by the newly entered number of points less 10 points and correcting TP to the nearest multiple consistent allowed potentiostat values for the TP parameter.

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Time per Point (TP)

Range:

273/273A	50 μs to 1074 s
263	120 μs to 1074 s
Versastat, 173 + 276	275 μs to 1074 s

Default: 0.1 s

Discussion: The Time per Point (TP) is just the period of time between the sampling of two data points. It cannot exceed the value of the Time of Step 1 (T1) and the program enforces that limitation. Enter the value in seconds (s) in multiples of 1E-6.

After changing TP, the Number of Points is updated based on the total time of the experiment and the new setting. Note that the Number of Points always includes 10 data points collected at the initial conditions before the first potential or current step.

Also note that if the Number of Points would exceed the maximum allowed, the program adjusts NP to the maximum and sets TP to the minimum value possible based on the total time of the experiment. The program may also adjust TP to make it consistent with potentiostat settings as well.

Time Step 1 (T1)

Range:

T1: TP to 3.221E6 s

Default: 10 s (T1)

Discussion: T1 corresponds to the duration of the waveform step. Since there would be no first step if T1 were 0, the minimum value you can assign to it is equal to the Time per Point (TP). Enter a value in seconds in multiples of 1E-4 s.

After T1 changes the program updates the Time per Point based on the total time of the experiment and the Number of Points less 10 data points, since the Number of Points always includes 10 data points collected at the initial conditions before the first potential or current step. If the new value of TP is less than its minimum allowed value, a "Step time too short" error message will occur.

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23 Chapter Twenty-Three Controlled Potential Coulometry

Overview

This chapter describes the parameters unique to a Controlled Potential Coulometry (CPC) experiment. These parameters define the CPC waveform. For additional setup information, refer to these chapters before or during the CPC setup:

Chapter 6 . . . . . setup menu
Chapter 13 . . . . . common parameters

Controlled Potential Coulometry Defined

In controlled potential experiments, the potentiostat steps the potential from the open circuit potential to a programmed potential(IP) while stirring the sample solution and monitors the current that flows. During the experiment experiment the software also displays an ongoing readout of the integrated current or charge. The response curve ideally displays an exponential current decaying versus time. This technique is useful for two reasons. For analytical determinations the total charge passed in the experiment is easily related to the concentration of the electroactive species in the cell. Alternatively, when dealing with a new compound, the total charge and the amount of material added allow calculation of the number of electrons transferred in the redox process.

The Pre-Electrolysis Time parameter allows for background electrolysis of the solvent before addition of your sample. Normally you would not add the sample until the pre-electrolysis time has passed and the experiment has begun. At the end of the pre-electrolysis time, the software records the background current and this background current is then subtracted form all subnsequent current measurements. This background current measurement and subtraction can be avoided altogether by setting the Pre-Electrolysis Time to Pass.

Initial Potential (IP)

Range:

263, 273/273A:	±10.000 V
Versastat, 173 + 276:	±8.000 V

Default: 0.000 V

Discussion: The Initial Potential is the potential that is applied at the start of the experiment and during the Pre-Electrolysis Time (CPC). Values are stored with a resolution of 1mV and the total potential range must be no more than 4 V. Otherwise a "Potentials out of range" error message will result. Also the total potential range during a scanning technique combines with the Scan Increment to

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determine Number of Points parameter. Thus adjusting the Initial Potential can also lead to a "Too many points" error message. You may enter the value vs. the open circuit potential by simply adding an 'o' or 'O' character at the end of the value.

Number of Points (NP)

Range: 1 to 3000 points

Default: 200

Discussion: The Number of Points(NP) is the total number of points taken during the experiment. For most of the experimental techniques, this is not user settable, but can be entered for the time based techniques, chronoamperometry, chronocoulometry, chronopotentiometry and controlled potential coulometry. For these experiments NP includes 10 data points collected at the initial conditions before the first potential or current step.

Note after you change NP, the program adjusts the Time per Point (TP) by dividing the total experiment time by the newly entered number of points less 10 points and correcting TP to the nearest multiple consistent allowed potentiostat values for the TP parameter.

Pre-Electrolysis Time (ET)

Range: 0 to 32,767 s, Pass

Default: 15 s

Discussion: The Pre-Electrolysis Time controls a variable delay during which the cell is on, the purge off and the stirrer on. Pre-electrolysis occurs at the Initial Potential and is intended to allow background electrolysis of the solvent before the experiment starts and before the sample of interest is added. After the program starts reporting the current and charge, the sample is added to the cell.

Time per Point (TP)

Range:

273/273A	50 μ to 1074 s
263	120 μs to 1074 s
Versastat, 173 + 276	275 μs to 1074 s

Default: 0.1 s

Discussion: The Time per Point (TP) is just the period of time between the sampling of two data points. It cannot exceed the value of the Time of Step 1 (T1) and the program enforces that limitation. Enter the value in seconds (s) in multiples of 1E-6.

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After changing TP, the Number of Points is updated based on the total time of the experiment and the new setting. Note that the Number of Points always includes 10 data points collected at the initial conditions before the first potential or current step.

Also note that if the Number of Points would exceed the maximum allowed, the program adjusts NP to the maximum and sets TP to the minimum value possible based on the total time of the experiment. The program may also adjust TP to make it consistent with potentiostat settings as well.

Time Step 1 (T1)

Range:
T1: TP to 3.221E6 s

Default: 10 s (T1)

Discussion: T1 corresponds to the duration of the first waveform step. Since there would be no first step if T1 were 0, the minimum value you can assign to it is equal to the Time per Point (TP). Enter a value in seconds in multiples of 1E-4 s.

After T1 changes the program updates the Time per Point based on the total time of the experiment and the Number of Points less 10 data points, since the Number of Points always includes 10 data points collected at the initial conditions before the first potential or current step. If the new value of TP is less than its minimum allowed value, a "Step time too short" error message will occur.

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Overview

This chapter describes the Model 270 iR Compensation feature. You use this feature to measure the uncompensated resistance in the cell between the reference and working electrodes. Normally, you would make the iR compensation measurement before running an experiment with another technique. This allows you to use the measured value as the iR compensation for a subsequent experiment.

iR Compensation Defined

Your experimental solution has a finite solution resistance associated with it and this is present between the reference and the working electrode. This solution resistance couples with the cell current to produce an iR potential drop that keeps the working electrode from being at the programmed potential.

The IR measurement technique allows you to use the program to estimate the uncompensated solution resistance and appropriately adjust the RU parameter. To do this you will have to select the IR mode parameter and set it to Measured. This setting is only available for IR Measurement Technique. When you return to your original experimental setup the IR mode will switch automatically to Entered and the measured RU value is preserved.

Note that the RU value measured and applied is specific to the current range selected. For this reason, you cannot select Auto for this technique. For best results, run your desired technique without IR compensation (IR mode = None) and with the current range set to Auto. Then set the current range to a fixed range consistent with the maximum current observed in your experiment. Finally switch to the IR measurement technique to actually estimate the uncompensated resistance. The advantage of this process is that the current range is set correctly before trying to measure the uncompensated resistance and you end up with an experiment performed both with and without IR compensation enabled, so that you can easily compare them afterwards.

To make the measurements here, set the Measure Potential to a potential where no redox processes occur. To begin with stick with the default values of the Pulse Height, Compensation Level and the Undershoot. Run the experiment. If the current overloads, decrease the Pulse Height and run it again. The program executes a number of cycles where it pulses the cell and observes the response. After each cycle it measures the undershoot value and compares it with the Undershoot parameter. If the measured undershoot is acceptable, the program adjusts the RU parameter upward. The experiment halts when either the Undershoot parameter is exceeded or the RU parameter reaches the product of the estimated uncompensated solution resistance and the Compensation Level.

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Discussion: Your experimental solution has a finite solution resistance associated with it and this is present between the reference and the working electrode. This solution resistance couples with the cell current to produce an iR potential drop that keeps the working electrode from being at the programmed potential.

The IR mode setting combines with the Uncompensated Resistance setting (RU) to allow you to adjust the applied potential values to correct for this problem using the positive feedback feature of the potentiostat. You may set this option to None for no compensation or Entered if you wish to use compensation. In that case you can use the RU parameter to enter an estimate of the solution resistance. As an alternative you may use the IR measurement technique to have the program estimate the solution resistance for you. When in the IR measurement technique, a third option of Measured is available which allows you to run an experiment to estimate RU for you. When you return to your original experimental setup the IR mode switches to Entered and the measured RU value is preserved.

Note that the RU value measured and applied is specific to the selected current range. Thus, you should avoid setting the current range to Auto when IR mode is set to Entered.

Measurement Potential (MP)

Range: ±10.000 V

Default: 0.000 V

Discussion: This is the potential at which the uncompensated solution resistance is estimated. Enter the value in volts, in multiples of .001. ***Note:*** You should set this parameter to a potential where no redox processes occur.

Operation

General: The purpose of an iR measurement is to establish a usable compensation value for a subsequent experiment using another technique. You can make the iR compensation measurement after selecting Measured or Entered. If you select Measured and press Run, the program will repeat the measurement until it finds the best compensation value for the entered compensation level and undershoot. If you select Entered and press Run, the program will make the measurement once to allow you to view the resulting current peak and change the entered compensation value, if desired. For both methods, the real-time current and potential values are displayed in a box at the lower left corner of the screen.

Setup: The first parameter selection to make is always the iR mode. When you select Entered, simply enter the desired compensation value at that time. If you select Measured, you must then specify the measurement potential, compensation level, undershoot, and pulse height before pressing Run to make the iR measurement.

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Example **85% Compensation, 100% Undershoot**

When you enter an 85% compensation level and a 100% undershoot value, the following sequence occurs:

1. The program takes an initial current sampling, calculates the actual uncompensated resistance, and displays a current response pulse representing no iR compensation. The program begins with a 0% compensation level so that it can approach the specified level slowly, while monitoring the undershoot percentage.

2. The program begins to repeat the sequence described in step 1, increasing the compensation level in increments of 5%, and displaying the successive pulses on the real-time plot. When it reaches the specified 85% compensation level, the measurement is over. The program plots the final current response pulse and displays the last value, which is 85% of the actual resistance, next to the Uncompensated Resistance parameter.

3. Note that the 100% undershoot value accepts any amount of undershoot and allows the iR measurement to be completed without special considerations. This is true because the undershoot will virtually never reach 100%, so a value of 100% tells the program to accept any amount of undershoot. Contrast this with second case below.

Example **85% Compensation, 10% Undershoot**

85% compensation and 10% undershoot are typical values, and we recommend them as good starting values. When you enter these values the following sequence occurs:

1. The program takes an initial current sampling, calculates the actual uncompensated resistance, and displays a current response pulse representing no iR compensation. The program begins with a 0% compensation level so that it can approach the specified level slowly, while monitoring the undershoot percentage.

2. The program begins to repeat the sequence described in step 1, increasing the compensation level in increments of 5%, and displaying the successive peaks on the real-time plot.

3. If it reaches the specified 85% compensation level, the measurement ends in the same way as the first case. However, if the undershoot percentage is more than 10% at any one of these iterations, the program reverts to the previous 5% increment and begins to increase the compensation in 1% increments.

4. When it reaches the compensation level where the undershoot is again 10%, the program reverts to the previous 1% increment and the measurement is over. The program plots the final current response peak and displays the last value, which is less than the specified 85% of the last calculated resistance, next to the Uncompensated Resistance parameter.

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Undershoot (UN)

Range: 1 to 100%

Default: 10%

Discussion: Oscillations in the applied potential can occur when using high Compensation Levels in iR compensation. The Undershoot parameter specifies how much oscillation in the applied potential you are will to tolerate, i.e. how much can the current reverse itself after a potential pulse. It is expressed as a percentage and represents ratio of the reverse current peak on the waveform to the initial current peak. When performing the IR measurement technique the program checks the measured undershoot after every cycle. If acceptable, the RU parameter is increased, otherwise it is backed off till the measured undershoot is less than Undershoot parameter.

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25 Chapter Twenty-Five Immediate Mode

Overview

Immediate mode allows you to control the cell directly without having to do an experimental setup. You can select the Immediate mode menu only when a potentiostat is connected to your computer. From the Immediate mode menu, you have direct control of the purge, stir, drop dispense, and rotating disk operations. You can also turn the cell on or off and apply a controlled potential or current to the working electrode.

Cell

Definition: Turning the cell on connects the cell counter electrode to the potentiostat, allowing current to flow in the cell. The current that actually flows depends on the cell chemistry and the controlled potential or controlled current applied to the working electrode.

Purpose: You may want to apply a continuous potential or current to the cell. You must turn the cell on to do this.

Warning! Turning the cell on with too high a potential or current applied to the electrode can be dangerous. It could result in harmful or lethal electrical shock or damage to the electrode. Be certain that you understand what will happen when you turn the cell on. Enter the value for the E Set or I Set parameter before turning the cell on and determine what effect the entered potential or current value will have on the cell.

Operation: Press the C key or click on the Cell box to turn the cell on or off from the Immediate mode menu.

Drop

Definition: When you turn Drop on, the potentiostat activates the Dislodge/Dispense line. If a Model 303A is connected to a Model 273, 273A, or 263, the potentiostat will send a dislodge/dispense pulse. When the Model 303A receives the pulse, it will knock the drop off the tip of the capillary and dispense a new drop.

Purpose: You may want to manually ensure a fresh drop before beginning an experiment or you may want to ensure proper capillary operation.

Operation: From the Immediate mode menu, press the D key or click on the Drop box to dispense a single new drop.

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selected Rotating as the electrode type from the Edit menu. The speed you enter will be added to the manual setting on the front panel of the Model 616 Rotating Disk Electrode. It will be in effect until you change it or select a new electrode type.

Purpose: You use this parameter to set the rotation speed for any experiment that requires a rotating electrode or to control a rotating electrode in Immediate mode.

Setup: When you select Rotating as your working electrode choice during an experimental setup, you must then go to the Immediate mode menu to enter a rotation speed. After you enter the rotation speed, you can go back to the edit menu to complete the setup or run the experiment.

Connections: The Model 273(A) controls the Model 616 through the Output connector. The rotation speed information is sent to the Model 616 through this front-panel BNC connector. Use a BNC to double-banana cable to connect to the Model 616 inputs labelled **Common** and **Input 1V/4k RPM**, located on the back of the Model 616 motor housing.

Operation: From the Immediate mode menu, press the R key to allow input. Then enter a rotation speed in rotations per minute. The speed will be the sum of the Model 616 manual setting and the value you entered from the Immediate mode menu.

Stir

Definition: When you turn Stir on, if you have a Model 273, 273A, or 263 potentiostat activates the stir line. If the potentiostat and it is connected to a Model 303A, the stirrer will be activated and will remain active until you turn it off. It does not come on automatically when you purge, as is the case when you purge from a technique menu.

Purpose: You turn the stir line on when you want the solution to be stirred continuously. For example, you may use Immediate mode to apply a continuous potential to the electrode to allow a species from the solution to adsorb onto the electrode. Stirring the solution during the adsorption keeps the species flowing to the electrode.

Operation: Press the S key or click on the Stir box to turn the stirrer on or off from the Immediate mode menu.

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Overview

The Autoexecute function allows you to set up and run a sequence of experiments without further operator intervention. While it is available for every potentiostat you are running, it applies only to the current cell.

The Autoexecute menu is accessible from two places in the Model 270 software. It appears on the New Technique pulldown menu as a technique that you can select and for which you can edit a setup. While an Autoexecute is running, it also appears as a choice on the Run banner menu. The menu choices available when you have selected it from the New Technique pulldown are: Get, Save, Run, Clear, Loop Begin, Total Cycles, and the filenames. If you are on the Run banner, the only available menu choice is Stop.

Get, Save, & Clear Autoexecute

These menu options allow you to save and recall Autoexecute setups to and from files and completely reset the existing setup to the default values. Note that Autoexecute assumes that all files will be stored in the Drive\Dir specified in the Defaults Files pulldown menu. You should not change the Files Drive\Dir setting after creating an Autoexecute file, as the program will not be able to execute it properly if it cannot find the setup files. If the program cannot find a setup file specified in the Autoexecute file, you will receive an error message when you select Run Autoexecute.

Input Setup and Output Data Filenames

Before it can run a series of experiments, Autoexecute requires you to designate a series of filenames. It needs both a setup filename for input and a data filename for for output for each individual experiment. Note that the setup files must exist before you can use them for autoexecution, or you will get a "File not found" error message when you attempt to run Autoexecute.

Also note that Autoexecute assumes that all files will be stored in the drive\directory specified in the Defaults Files Drive\Dir menu option. You may not specify a Drive\Dir with the filename and filenames are limited to 12 characters.

Likewise, you should be cautious about specifying data filenames that already exist. If you specify an existing file, the software will request that you confirm overwriting the file. A setup or data filename can be accessed by clicking on the menu option as usual, or by typing the hot key string. For example, type O5 to change the output data filename for the fifth experiment. You may enter up to ten filenames for both setup and data.

The sequence of experiments terminates when the program encounters a setup file or a data file that is blank. Thus, entering setup filenames for the first, second, and fourth setup files only would cause the fourth setup to be ignored when the

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selected, Stop will abort the whole sequence of experiments in the Autoexecute series. However, any data that is already stored on disk remains on disk for you to examine.

You can determine the status of the Autoexecute function by selecting autoeXecute while an autoexecute is in progress. The bottom status line will display the Step & Loop numbers in addition to the current setup file being used.

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27 Chapter Twenty-Seven Cut/Paste Operations

Overview

The Cut/Paste feature of the software allows the user to store data, calculation results, and experimental parameters in a simple ASCII text file. This is in contrast to the Export Data menu option on the File banner menu which lets you export only *x,y* data pairs. These numbers, whether from Cut/Paste or Export Data, can then be easily imported into other programs such as spreadsheets. Additionally, cut/paste files can be re-imported into the Model 270 software, allowing you to create custom graphs of your experimental results.

Using the Cut/Paste feature requires first deciding what information you want in the file and actually setting it up using the Cut/Paste menu accessed on the File pulldown menu. Once a cut/paste file has been opened for input, you can use the Free Cursor, Curve Cursor, Area, Peak, Half Width and Line Fit menu options to locate data or results. After the data or results are displayed, using the F2 or C key will cause the software to store the information in the cut/paste file.

In many cases, you will want to cut/paste results or data from a number of different experiments into a single cut/paste file so you can see the results of varying an experimental parameter. In a later example, we will examine the effect on peak current produced by varying concentrations of the sample of interest.

Cut/Paste Menu

The Cut/Paste *selection* on the File menu allows you to set up the cut/paste file before you actually perform the cut/paste operations. When you select Cut/Paste from the File menu, the Cut/Paste Setup menu appears. This section describes how to use the selections on the Cut/Paste Setup menu to set up the file.

Note that there is only one cut/paste file active at a time, and it stays available for more results from one experiment to the next until you specify a new file name from the Cut/Paste menu.

When you select the Cut/Paste menu and set up the file, the setup is automatically recorded in memory as soon as you make your selections. Any data you cut/paste later is automatically stored as a line of five numbers in the same file as soon as you press the F2 key.

File structure: Each numerical entry in the cut/paste file contains five fields, with each field separated by a delimiter. The delimiter, defined in the Files pulldown on the Defaults banner menu (see Chapter 10), is a comma by default but tab, space, or semicolon characters may be specified instead.

The following paragraphs describe the fields in the order in which they are stored in the file:

- **Field 1:** The first field contains the point number, which is only meaningful when you do a cut/paste operation with the curve cursor, or cut/paste the peak

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For example, in a series of cyclic voltammetry experiments, you can use UE to reserve the first parameter field for the concentration peak heights, which you would use as an independent variable. You could then use the Peak Menu option to create a file containing peak heights results as a function of concentration. For subsequent reference, you can use the Comment selection on the Cut/Paste menu to describe what the parameter 1 value represents, or use parameter 2 as a mini-comment with UE.

If you do not plan to use the first or second parameter field, select NU (Not Used), the default setting for both fields.

- **Comment:** This selection allows you to enter a comment into the cut/paste file. The comment lines are useful for describing the parameter fields and the purpose of the experiment. You can enter up to 80 alphanumeric characters as a comment. Any standard keyboard character is allowed. You may also enter multiple comments if you wish to store more than one line. Comments are immediately stored in the cut/paste file whenever you enter one.

Cut/Paste Output

The values stored in a cut/paste file are summarized in the following table. Note that Peaks and Limits are like normal curve cursor entries with a point number followed by *x* and *y* values.

Menu Option	Information Displayed	1st Field	2nd Field	3rd Field
Curve Menu				
Area, Pk, & Half-Width	Area	-32765	Area	0
Half-Width		-32757	Half-Width	0
Curve Cursor	Data Point	Pt.#	X	Y
Free Cursor	Cursor Location	-32767	X	Y
Draw Line	Slope/Intercept	-32766	Slope	Intercept
Line Fit	Slope/Intercept	-32764	Slope	Intercept
	% Errors	-32763	Std Dev of Slope	Std Dev of Intercept
	Statistics	-32762	χ^2	Correlation

Using Cut/Paste

In the following example, you will use a series of data files from the sample data disk to illustrate the effect of scan rates on peak heights of Pd+2 in cyclic voltammograms. In doing so, you will set up a sample cut/paste file, selectively store corrosion rates in it along with a user-entered parameter (concentration of ferrocene), and then re-import the data to produce a plot of vs concentration.

First, set up the cut/paste file:

1. Select File on the Main banner menu.

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13. To specify what information to import, change to the Defaults banner menu and select the Files pulldown. Then choose X import field # and pick 4th field, since scan rate was the 1st parameter which makes it the fourth number on a Cut/Paste line. Then choose Y field and pick 3nd field, since current ends up as the third number on a Cut/Paste line. If you wanted these settings to persist between sessions with the software, you would need to use Save on the Defaults banner menu to save them in the DEFAULTS.270 file.

14. You are now ready to re-import the data. Select Import Data from the File pulldown menu on the Main banner. Then, type sample.txt in response to the prompt for the file name. If all goes well, the program will inform you that it imported three data points and then graph them.

The point of this example was to illustrate one possible use of the Cut/Paste feature, so we will stop here. If this were your own data, however, you might wish to manipulate the data before re-importing it. This is possible using many of the commercially available spreadsheet programs to read in the Cut/Paste file. After manipulation of the data in the spreadsheet, you could save it as a similar ASCII file for importation back into the 270. In the example above, this would allow you to plot I vs $v^{1/2}$, which would be a straight line for a solution based redox couple in cyclic voltammetry. After re-importation, all of the 270's capabilities are accessible to you, so you could add a title and meaningful labels to plot, perform a a linear squares fit of the plot and then print it out for publication. In any case, the power of Cut/Paste along with the Import Data feature should now be more obvious to you now.

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A Appendix A Menu Structure

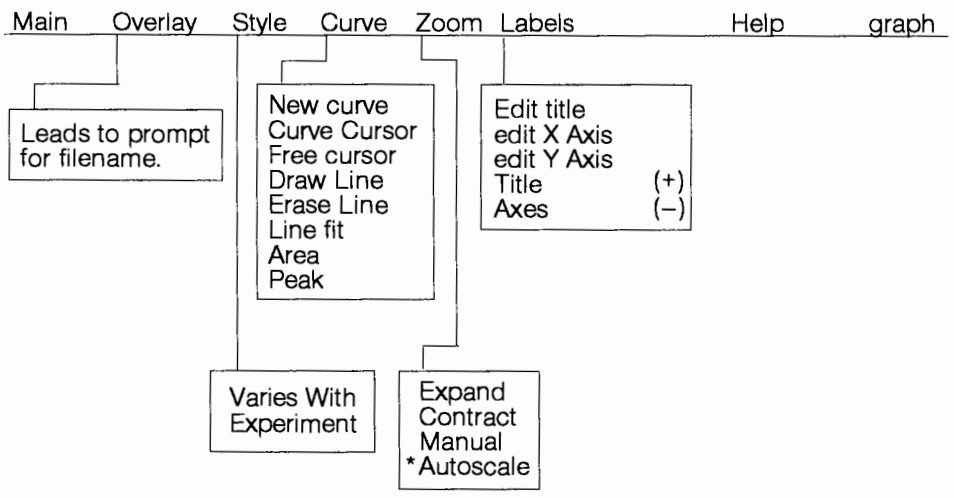
Overview

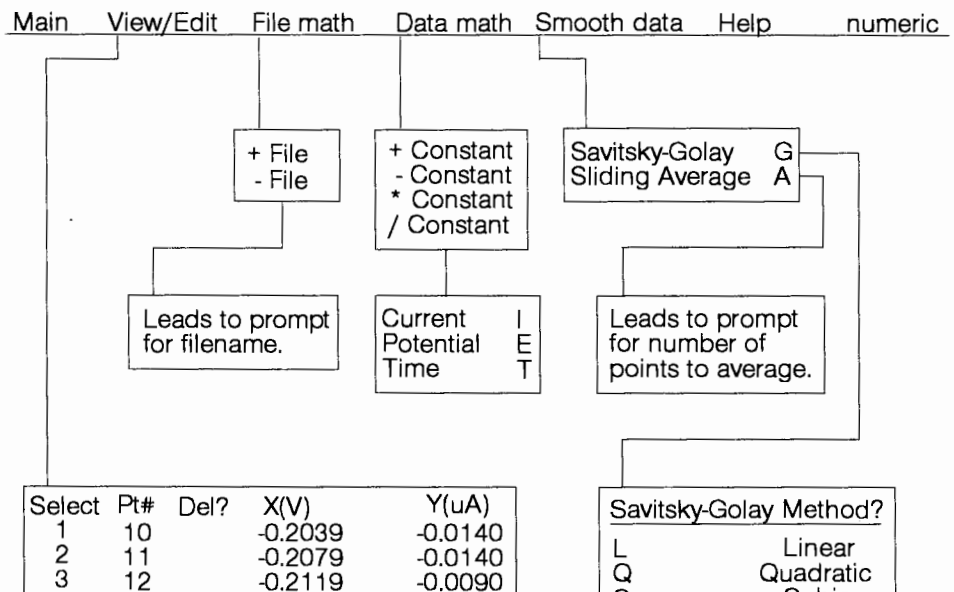
To simplify the user's understanding and operation of the Model 270 Software, it is organized into a series of functions accessed from menus and submenus. These menus and the functions they make available are logically organized in a tree-like structure that allows the user to easily branch to any part of the program and perform the desired operations. The organizational hierarchy of the banner menus, submenus, and pulldowns is shown on the following pages.

Please note that there are often several paths through the menu structure that reach the same selection. Also, the function keys provide shortcuts to many operations (see Chapter 4).

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MODEL 270 MENU STRUCTURE





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B Appendix B Model 270 File Formats

Overview

A Model 270 data file consists of two parts: (1) a header describing the experiment; and (2) a data section containing the numeric data. The header is always in ASCII (American Standard Code for Information Interchange). The data field format can be either ASCII or binary. (See "Examining or Editing a Data File" on the next page for a caution about editing binary files.) Use this procedure to select the desired data format:

1. From the Defaults banner menu, select Files.
2. Select Save Mode.
3. Select either ASCII or Binary.

Header

The header contains all the parameters that appear in the Edit pulldown menu, as well as some additional information. Each line of the header begins with a two- or three-character mnemonic, the same as used on the Edit pulldown, that identifies the parameter given. Additional mnemonics not used on the Edit pulldown are listed below. In each line, the mnemonic is followed by the full name of the header entry (in parentheses) and a description of the entry.

PN (Program Name): Model 270.

FV (File Version): Version number of the Model 270 software that stored the file.

EXT (Experiment Type): The experiment type (using the same abbreviations that appear on the New Techniques menu), and the experiment name.

DAT (Data Type): The data file format (Binary or ASCII). The program uses this field to determine how to read data into the file. For setup files, this line is set to Setup.

FLS (File Status): The edit status (Edited or Normal). This field lets the program know if the data has been edited or not.

DR (Date Run): The date the experiment was begun.

TR (Time Run): The time the experiment was begun.

PC (Parameters Collected): The type of data collected: I E T (for current, potential, and time).

DE1: User-entered comment.

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Since each sampling involves an applied potential and a current response with respect to time, three numbers are associated with each data sampling. Thus, each data file will store potential, current, and time values for each sampling as separate but contiguous blocks of data. All three values are stored as IEEE four-byte real numbers. Be sure that any program, compiler, or interpreter you use supports this format.

For example, if the value for number of points is 1000 (NP 1000), 1000 four-byte real numbers are stored for current data, followed by 1000 four-byte real numbers for potential data, followed by 1000 four-byte real numbers for time data. The data field for 1000 data points for any one of these three parameters will occupy 4000 bytes (1000 points at four bytes per point). The entire data field for all three stored values for 1000 points will occupy 12,000 bytes.

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C Appendix C GPIB Addresses and Problems

Overview

This appendix describes how to set addresses on the GPIB switches of the various potentiostats that can be used with the Model 352 SoftCorr II software. It also includes a troubleshooting procedure for "Potentiostat not found" errors.

The GPIB address you set on your potentiostat must agree with the address set on the Assign Cells selection on the Defaults Experiment pulldown menu (see Chapter 10). The potentiostats are shipped from the factory with the GPIB address set to 14, which is also the default setting for Cell #1 in the software. If you change the potentiostat address you must also reset the software address.

If you are using more than one potentiostat, you must set them to different GPIB addresses to make simultaneous use of them with the Model 352 software.

GPIB Address Switch Settings

Setting the GPIB address on a potentiostat is not difficult. The address is set with DIP switches on the potentiostat for the Models 273, 273A, 173/276 and the Versastat. For the Model 263 you use the front panel.

On the Models 273 and 273A the DIP switches are on the back of the instrument just to the left of the IEEE-488/GPIB INTERFACE connector. The switches you are concerned with are labeled GPIB ADDRESS.

With a VersaStat, you need a screwdriver to remove the four screws that hold the top cover on. The GPIB switches are in the middle of the main circuit board. **Be sure to power this instrument down and remove its power cable before opening its case.**

On a Model 173/276, the Model 276 must be removed. **Be sure to turn off the power to the Model 173 before you remove the Model 276.** Then, simply unscrew the small knob on the bottom of the Model 276 front panel that holds it in place, and pull the Model 276 out. The GPIB switches are toward the bottom back edge of the digital board.

To change a switch setting, press the rocker down on the side you wish to set it to. For example, pressing it down on the OFF or 0 side sets that switch to OFF.

All potentiostats are shipped from the factory with GPIB switches 2, 3, and 4 set to ON, and all others OFF. **Switches 6, 7, and 8 will always be set to OFF**, while switches 1 through 5 control the GPIB address of the potentiostat with binary weighting of the switch. If all five switches are OFF, the address is 0. Avoid this setting—the National Instruments GPIB card uses it.

If only switch 1 is ON, the address is 1. If only switch 2 is ON, the address is 2; if only switch 3 is ON, the address is 4; if only switch 4 is ON, the address is 8; if

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and press <Enter>.

(b) Look for a line like:

DEVICE=C:\subdir\GPIB.COM

The "subdir" in this line should be the name of the utility subdirectory that you specified when installing the software. The default subdirectory in the Install program is c:\parc\. If the directory is c:\PC-GPIB\ or c:\GPIB-PC\, then you probably used the National Install program after the EG&G PARC Install program. Unfortunately, the default GPIB.COM file configuration from National does not work with EG&G PARC software unless extensive work is done with the IBCONF.EXE configuration program. If you don't find a line like this, then DOS is not even trying to load the GPIB.COM device driver. The solution in both cases is either reinstalling the software or editing the CONFIG.SYS file directly to add this line.

(c) Now, get a directory for this device driver. Type the following:

DIR C:\subdir\GPIB.COM <Enter>

where "subdir" is the name of the subdirectory you found in the CONFIG.SYS file. You should see DOS display the GPIB.COM file's size, time, and date of last modification. If you get a "File not found" error message, the GPIB.COM file is not being properly loaded because it is not in the subdirectory specified in CONFIG.SYS. (This could happen if you had used a utility program to rename the subdirectory your software is installed in). The solution is either to copy the GPIB.COM file to where the CONFIG.SYS file says it is or to edit CONFIG.SYS to make it specify the real directory.

If, after working through this list of possible GPIB problems, you are still having troubles, feel free to call in and ask for EChem Technical Support. The phone number is (609) 530-1000. It would also be helpful to have printed copies of your CONFIG.SYS and AUTOEXEC.BAT files as well as a printed directory listing of your EG&G PARC software subdirectory. As an alternative, you can fax us the same information along with a description of the problem at (609) 883-7259.

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D Appendix D Files Included on Distribution Disks

Overview

The Model 270 Research Electrochemistry software is provided on two sets of floppy disks to accommodate a wide range of IBM-compatible personal computers. One set consists of five 360 Kbyte, 5-1/4 inch disks, the other of three 720 Kbyte, 3-1/2 inch disks.

Both sets contain identical files, but fewer 3-1/2 inch disks are required as more files fit on each disk. The files provided are grouped below according to their locations on the 5-1/4 inch disks. Please note that all files listed below for the Model 270 Distribution Disk and the Model 270 Utility Disk are grouped together on the 3-1/2 inch Model 270 Distribution & Utility Disk. Also, all files listed below for the Install Drivers Disks #1 and #2 are grouped together on the 3-1/2 inch Install Drivers Disk.

If you have purchased an EG&G PARC Model 270 system, all software has been installed by the factory. The Model 270 software disks are provided as a backup in case of emergency.

Before continuing, take a moment to fill out the Software License Agreement and Warranty Registration card provided. By returning this card, you are assured of being informed of software updates and enhancements as they become available.

Files Included on the Model 270 Distribution Disk

M270ZIP.EXE	Self-extracting ZIP file. Contains M270.EXE and M270.270 overlay file.
README.270	Readme file with any last-minute changes.
VERSION.270	Version number of M270.EXE.
LABEL.TXT	Text file used by labeling utility.

Files Included on the Model 270 Utility Disk

DEFAULTS.270	Model 270 Defaults banner settings.
HELP.270	Help text and Help index files used by Model 270.
HELPINDX.270	
README.270	Readme file with any last-minute changes.
VERSION.270	Version number of M270.EXE.
LABEL.TXT	Text file used by labeling utility.

Files Included on the Model 270 Data Disk

PDCV1.DAT	Cut/Paste tutorial sample data files.

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Electrochemistry Software Problem Report Form

Name _____ Phone _____

Company _____ Fax _____

Address _____

_____

Please circle or fill in the blanks with the appropriate information about how your system is configured.

Software M270/250 M271/COOL M352/252 M398 Headstart Version _____
Other _____ Willing to try beta version? Yes No

Computer Brand _____ Model/CPU _____ Speed _____ DOS v. _____

Potentiostat M273A M273 M263 M253/Versastat M173/276 ROM v. _____
Option Boards _____ Lockin/FRA _____

GPIB Card PC2 PC2A PC2/PC2A PC-AT MC-GPIB GPIB.COM v. _____

Other

Does the computer lock up?	Yes	No
Does the program terminate to a DOS prompt?	Yes	No
Are copies of your CONFIG.SYS & AUTOEXEC.BAT files attached?	Yes	No
Are data or setup files supplied?	Yes	No

At Princeton Applied Research, our goal is high quality software. If you encounter a problem, we want to know about it. Please give us a detailed description below. Use the back of the form if you need more space. *If your problem concerns a faulty experimental setup, please enclose a setup file, so that the problem can be reproduced. If the problem deals with calculations, please enclose the data file you used.*

Return completed forms to: Dr. Matthew Rhodes, Electrochemist/Programmer, EG&G Instruments, Princeton Applied Research, P.O. Box 2565, Princeton, NJ 08543. Or fax to 609-883-7259.

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Electrochemistry Software Suggestion Form

Name _____ Phone _____

Company _____ Fax _____

Address _____

_____

Software Program

Circle one: M270/250 M271/COOL M352/252 M398 Version _____

Other _____ Willing to beta test? Yes No

At Princeton Applied Research, we want to know what you think about our software. Is there some feature missing or some quirk that drives you up the wall? Let us know. Just give us a description of the features you want or of what needs improvement. Use the back of this form for more space or to sketch how the software might look. We cannot promise to implement everything requested, but we do promise to use your suggestions to decide what features go into our next releases.

Return completed forms to: Dr. Matthew Rhodes, Electrochemist/Programmer, EG&G Instruments, Princeton Applied Research, P.O. Box 2565, Princeton, NJ 08543. Or fax to 609-883-7259.

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Warranty

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EG&G Instruments reserves the right to make revisions to the *Model 270/250 Electrochemical Analysis Software* program(s) at any time without incurring any obligation to install same on software previously purchased.

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SHOULD YOUR EQUIPMENT REQUIRE SERVICE

A. Contact the Service Department (609-530-1000) or your local representative to discuss the problem. In many cases it will be possible to expedite servicing by localizing the problem to a particular plug-in circuit board.

B. If it is necessary to send any equipment back for service, we need the following information.

1. Model number and serial number.
2. Your name (instrument user).
3. Your address.
4. Address to which the instrument should be returned.
5. Your telephone number and extension.
6. Symptoms (in detail, including control settings).
7. Your purchase order number for repair charges (does not apply to repairs in warranty).
8. Shipping instructions (if you wish to authorize shipment by any method other than normal surface transportation.)

C. **U.S. CUSTOMERS** - Ship the equipment being returned to:

EG&G PARC
375 Phillips Blvd.
Trenton, NJ 08618

D. **CUSTOMERS OUTSIDE OF U.S.A.** - To avoid delay in customs clearance of equipment being returned, please contact the factory or the nearest factory distributor for complete shipping information.

E. Address correspondence to:

EG&G PARC
P.O. Box 2565
Princeton, New Jersey 08543-2565

Phone: (609) 530-1000
TELEX: 84-3409
FAX: 883-7259

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Model 270/250 Research Electrochemistry Software User's Guide (222153-A-MNL-A) Changes in Software Version 4.10

This addendum describes the improvements incorporated in Version 4.10 of the Model 270/250 software and Version 1.10 of the COOL Kinetic Analysis Software (which is supplied with the Model 270 software). We have indicated in parentheses the places in the *Model 270/250 User's Guide* and *Model 271 COOL User's Guide* where information is clarified or superseded by this addendum. In addition to the new features described below, we have added various minor enhancements and have improved the overall reliability of the software.

The addendum also includes some selected references to the literature on the COOL algorithm, mechanistic models, and electrochemistry in general.

New Features in Model 270/250 Software

Version 4.10 of the Model 270/250 software includes the following enhancements:

Microsoft Windows 3.1 Support

Sample PIF (ECHEM.PIF) and icon (ECHEM.ICO) files are copied to the Model 270 program directory along with the Model 270/250 program files during installation. The PIF file is set up to run the Model 270/250 software from Windows in full-screen mode.

AUX A/D Data Collection

You can now collect additional data using the AUX A/D Input connector on the back of a Model 273A potentiostat, or a Model 263A with the /98 option installed. This would allow you to correlate a voltage signal with an electrochemical experiment. For example, you could monitor the voltage across a thermocouple to provide an indication of temperature during an experiment. Or you could use a QCA-917 Quartz Crystal Analyzer with the potentiostat to correlate mass and viscoelasticity changes in a solution or coating with electrochemical activity. More information on the use of the Quartz Crystal Analyzer is given later in this addendum.

AUX A/D data can be collected concurrently with all techniques except IR Compensation. You can plot the AUX data you collect versus t, versus I, or versus E.

Raw Data Factors

You can now multiply raw I, E and AUX Input data by some factor you define. This could be used to scale the raw AUX data values to user-defined units. For example, you could convert raw AUX data to temperature units.

Note: Default factors for I, E and AUX Input data are set to 1.0.

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Multi-CV Data Collection

The software now allows storage of all data cycles during a multiple-cycle cyclic voltammetry experiment. When the store cycle (SC) equals All (pp. 78-79):

- 2–256 cycles (NC) can be collected.
- Only Acquisition Mode (AM) 4/4 and All are allowed.
- When the second vertex potential (V2) is set to Pass:
  - The Final Potential (FP) is "locked" to the Initial Potential (IP).
  - The Vertex Delay (VD) is not allowed.
  - The waveform cycles between IP (which is also FP) and Vertex 1 Potential (V1).
- When the second vertex potential (V2) is set to a potential:
  - The Final Potential (FP) can be different from the Initial Potential (IP).
  - The waveform ramps from IP to V1 then cycles between V1 and V2 and then ramps from V2 to FP.

Battery Cycle Testing/Decision Making Support

We have added a Stop On (SO) setup parameter for the simple step experiments: chronoamperometry (CA), controlled potential coulometry (CPC), chronocoulometry (CC), and chronopotentiometry (CE). This parameter allows automatic termination of the experiment when one of the measured data values (*I*, *E*, or *Q*) climbs above or falls below a user-entered maximum or minimum value.

The following lists the setup menu support for SO in each technique:

I > #A (CA and CPC techniques)
I < #A (CA and CPC)
Q > #C (CC and CPC)
Q < #C (CC and CPC)
E > #V (CE)
E < #V (CE)

Note: The Maximum Potential (ME) parameter in chronopotentiometry has been replaced with SO.

Automatic Data Saving

The software, after it has completed data collection for an experiment, will now automatically save the data to protect it from unexpected mishaps such as power outages. The data is saved to a file named TEMPFILE.270 in your Model 270/250 program directory. This file can be recalled as a standard Model 270/250 data file.

Note that this new automatic save feature does not imply that you never have to save your data. When the data file is saved, the parameters used to set up the experiment are saved with the data. Whenever you make changes to the setup or perform an operation on the data, it is important to save the data file again so the changes will not be lost. You will also want to save the final results of your experiment at the end of your operating session, before you exit the program.

The program does not save data files automatically during an Autoexecute sequence.

Omitting and Restoring Points

The options to graphically Omit and Restore points on a plot have been added to the Graph banner Curve pulldown menu (pp. 32-36). The Omit Points and Restore Points features allow you to select a portion of the data to be deleted or undeleted. You can use either the mouse or the cursor keys to position the crosshair cursor on the data curve in the same manner as the Curve Cursor. The left and right mouse buttons or the "b" and "e" keys can be used to set the beginning and ending points of the data to be selected.

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Omitting and Restoring Points

The options to graphically Omit and Restore points on a plot have been added to the Graph banner Curve pulldown menu (pp. 52-55). These features also have been added to the Model 270/250 software (see page 3 of this addendum for an explanation).

Installation

You must use the Install disk to install this new version of the software on your computer. Just insert the Install disk in the drive, type **install**, and answer the questions.

Errata

In the *Model 271 COOL User's Guide*, the discussion of the Quit function on page 48 contains several errors. The second paragraph in the Quit section should read as follows:

> If you have unsaved data, the program will ask you whether you want to save it. If you answer "yes" you will be prompted for a file name before you are allowed to exit.

Please delete the other material in the paragraph.

COOL Literature References

For your convenience we have compiled the following list of references to the literature on the COOL algorithm and mechanistic models.

General References on COOL Algorithm

Osteryoung, J. "Inference of Mechanism from Kinetic Analysis of Pulse Voltammetric Data," *Chemometrics & Intelligent Laboratory Systems* **1991**, *10*, 141-154.

O'Dea, J.; Osteryoung, J.; Lane, T. "Determining Kinetic Parameters from Pulse Voltammetric Data," *J. Phys. Chem.* **1986**, *90*, 2761-64.

O'Dea, J. J.; Osteryoung, J.; Osteryoung, R. A. "Square Wave Voltammetry and Other Pulse Techniques for the Determination of Kinetic Parameters. The Reduction of Zinc(II) at Mercury Electrodes," *J. Phys. Chem.* **1983**, *87*, 3911-3918.

Specific References on Mechanistic Models

O'Dea, J. J.; Wikiel, K.; Osteryoung, J. "Square-Wave Voltammetry for ECE Mechanism," *J. Phys. Chem.* **1990**, *94*, 3628-3636.

Murphy, M. M.; O'Dea, J. J.; Arn, D.; Osteryoung, J. G. "Theory for Cyclic Staircase Voltammetry for First-Order Coupled Reactions," *Anal. Chem.* **1990**, *62*, 903-909. (CV calculations for EC, CE, and ECat mechanisms.)

Zeng, J.; Osteryoung, R. A. "Square Wave Voltammetry for a Pseudo-First-Order Catalytic Process," *Anal. Chem.* **1986**, *58*, 2766-2771. (SWV calculation for ECat mechanism.)

O'Dea, J. J.; Osteryoung, J.; Osteryoung, R. A. "Theory of Square Wave Voltammetry for Kinetic Systems," *Anal. Chem.* **1981**, *53*, 695-701. (SWV calculations for QRev, CE, EC, and ECat mechanisms.)

Christie, J. H.; Turner, J. A.; Osteryoung, R. A. "Square Wave Voltammetry at the Dropping Mercury Electrode: Theory," *Anal. Chem.* **1977**, *49*, 1899-1903. (SWV reversible calculation.)

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