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EG&G

Model 263A

Potentiostat/Galvanostat

User's Guide

222688-A-MNL-E

EG&G INSTRUMENTS, INC.
Princeton Applied Research

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FCC Notice

This equipment generates, uses, and can radiate radio-frequency energy and, if not installed and used in accordance with this manual, may cause interference to radio communications. As temporarily permitted by regulation, operation of this equipment in a residential area is likely to cause interference, in which case the user at his own facility will be required to take whatever measures may be required to correct the interference.

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Chapter 1
Introduction

1.1 OVERVIEW

The EG&G Princeton Applied Research Model 263A Potentiostat/Galvanostat is a powerful and versatile instrument for the electrochemistry researcher. It can be controlled either directly from its front panel or from a personal computer.

This instrument uses the latest design advances to provide high performance, ease of use, and great versatility in electrochemical measurements. Its ±20 V compliance and ±200 mA *(±2 A with 94 Option)* output capability allow rapid and accurate potential or current control in virtually any electrochemical cell. Component selection, shielding, grounding, and circuit design are all carefully optimized for minimum internal electronic noise, giving high sensitivity and a quiet output signal. Low-pass filters virtually eliminate noise arising in the cell itself.

A high-performance current-to-voltage converter circuit is included for accurate, rapid, low-drift current measurements, free of degradation from cell-cable resistance, capacitance, and inductance. The new self-calibration feature assures the accuracy of your data, while BNC connectors conveniently placed on the front panel make all signals of interest available for analog recording.

1.2 CONTROLLING THE MODEL 263A

Front-Panel Control

You can conduct a wide variety of static or scanning experiments directly from the front panel of the Model 263A. Its controls have been ergonomically designed for ease of use and maximum flexibility.The large, convenient parameter input knob, pushbuttons, and alphanumeric liquid crystal display streamline the process of setting up your experiment.

After the experiment begins, the display panel presents continuously updated information on its progress, including the parameters in effect and the cell voltage and current. A simple touch of a button implements such advanced features as automatic current ranging and current-interrupt iR compensation.

Remote Computer Control

For even greater versatility, you can control the Model 263A from an IBM-compatible personal computer via an IEEE-488 (GPIB) or RS-232 interface. This enables you to take advantage of the EG&G Electrochemical Command Set, a group of over 100 mnemonic software instructions that make it easy to create your own experiments. The Electrochemical Command Set, which is built into the Model 263A's read-only memory, has been specifically developed for research electrochemistry, dc corrosion measurements, and electrochemical impedance measurements.

These commands place unprecedented flexibility in the hands of the electrochemist. They provide:

- Access to most Model 263A front-panel functions.
- Control of all timing functions.
- Application of pulse and staircase waveforms.
- Automatic acquisition of data with or without current auto-ranging.
- Data averaging in real-time.
- Internal storage and arithmetic data manipulation on your lab computer.

These commands and all necessary instructions for operating the Model 263A from an external computer are described in the separately bound *Model 263A Command Set Handbook*.

The EG&G PARC HeadStart™ Creative Electrochemistry Software – included free with the Model 263A – provides a simple way to access the Electrochemical Command Set. HeadStart is easy to use and runs on any IBM PC or fully compatible personal computer. To learn how to use it, refer to the *HeadStart* instruction manual included with the Model 263A.

The Model 263A is also completely compatible with the standard EG&G PARC electrochemical software. So you can also run your experiments with any of our dedicated software packages, such as the Model 270/250 Research Electrochemistry Software, the Model 398 AC Impedance Software, or the Model 352 Corrosion Measurement and Analysis Software, on an IBM PC or fully compatible computer connected to the Model 263A.

When controlled by HeadStart or any of the standard EG&G PARC electrochemical software, the Model 263A can turn on and off the Model 616 Rotating Electrode System or send Purge, Stir, and Dispense signals to the Model 303A Static Mercury Drop Electrode.

The Model 263A is fully equipped to conduct experiments controlled from your computer. It includes:

- Two 14-bit digital-to-analog converters (BIAS and MODULATION) for versatile waveform generation. ***Note:*** *In units having 91 Option, MODULATION DAC is 16 bits.*
- A 12-bit analog-to-digital converter to measure current and potential.

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- An on-board microprocessor to perform the experiment defined by the Command Set.

- On-board memory to store the programmed parameters and data point values.

When the programmed experiment is finished, the data can be transferred to the computer for plotting or further processing.

1.3 OPERATING MODES

Potentiostatic
In this mode, the Model 263A controls the potential at the working electrode with respect to the reference electrode. The potential at the counter electrode is driven to the potential required (consistent with the ±20 V compliance of the control amplifier) to establish the desired working electrode potential. The range over which the working electrode potential can be controlled is ±10 V, although only a ±2 V range can be used in a single experiment. *This range is extended to ±8 V with Option 91.*

Galvanostatic
In galvanostatic operation, the Model 263A controls the current between the counter and working electrodes at the specified fraction of the selected current range (up to the maximum of two times the current range). The counter electrode is driven to the potential required (consistent with the ±20 V compliance of the control amplifier) to establish the desired cell current. The reference electrode is not used in the control loop, but is usually used to measure the potential at some point in the electrochemical cell.

1.4 ABOUT THIS MANUAL

This *User's Guide* provides details of the physical and electrical characteristics of the Model 263A, and describes how to operate it as a stand-alone instrument controlled from its front panel. Instructions for operating the unit remotely from a personal computer or workstation, via either the RS-232 or GPIB (IEEE-488) interface port, are given in the separately bound *Model 263A Command Set Handbook*. The *HeadStart Creative Electrochemistry Software Instruction Manual* shows you how to use HeadStart.

The *Model 263A Command Set Handbook* gives, in addition to the command descriptions, detailed explanations of GPIB (IEEE-488) and RS-232 communications and how to set the communications parameters. It also includes an application note on waveform programming, one of the most useful functions of the Model 263A.

This *User's Guide* is organized into eight chapters and one appendix. Chapter 1 provides a general description of the Model 263A. Be sure you understand the information in Section 1.5 about the polarity convention used in this instrument.

The appendix describes the physical and electrical characteristics of the instrument. It includes a description of the internal organization of the instrument and its electrical circuitry. Pinout descriptions of the connectors are also provided.

Chapter 2 describes recommended safety precautions for operating this instrument, including the provision of adequate ventilation. It also tells how to deal with transients on the power line, and discusses the unlikely possibility of the instrument causing radio frequency (RF) interference.

Chapter 3 describes the functions of the front- and rear-panel connectors, and shows how to connect the Model 263A to the test cell, pen recorder, and other equipment you may wish to use with it. Chapter 3 also explains how to set the instrument for operation with different input power voltages, replace the power line fuse, and determine whether power is being applied to the cell cable.

The Model 263A may be mounted in a standard 19-in. (47.5 cm) equipment rack assembly. Chapter 3, Section 3.4 provides mounting instructions.

After you inspect the Model 263A for shipping damage, but before you begin to use it for your work, run the sample session in Chapter 4 to ensure that it operates correctly. The sample session will also introduce you to the operation of the instrument.

Chapter 5 describes the functions of the controls, liquid crystal display, and indicator lights on the front panel. It then shows you how to set up the basic experiment parameters that are common to both potentiostatic and galvanostatic operation and seldom require changing. It also shows how to set the GPIB and RS-232 communications parameters necessary for controlling the instrument from a host computer.

After you have set these parameters, you must also set the parameters that are specific to the kind of experiment you wish to do. To complete your preparation and run your experiment, continue to either

- Chapter 6 to set potentiostatic parameters and run a potentiostatic experiment, or

- Chapter 7 to set galvanostatic parameters and run a galvanostatic experiment.

Certain conditions that interfere with normal operation will cause error messages to be displayed on the LCD panel. These messages are explained in Chapter 5, Section 5.10.

Chapter 8 is an alphabetical list of all the experiment parameters that can be set from the front panel of the Model 263A, with detailed explanations included for each.

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1.5 POLARITY CONVENTION

The Model 263A follows the American polarity convention and the display indications are consistent with that convention. Positive current is cathodic, that is, a current is defined as positive if reduction is taking place at the working electrode. Negative current is anodic, that is, a current is defined as negative if oxidation is taking place at the working electrode.

In potentiostatic operation, making the applied potential more positive will cause the current to become more anodic. Conversely, making the applied potential more negative will cause the current to become more cathodic. This is true for all potential sources at the external input (EXT INPUT) connector.

In galvanostatic operation, making the applied potential more positive by any means except applying a potential to EXT INPUT will tend to make the current more cathodic. Making the applied potential more negative will tend to make the current more anodic. *This sense is reversed at the EXT INPUT connector. There, making the input more positive will make the cell current more anodic. Making the input more negative will make the cell current more cathodic.*

Bear in mind that EXT INPUT is a high-impedance (100 kΩ) input in both potentiostatic and galvanostatic operation. (In galvanostatic operation, 1 V applied results in a full-scale current, assuming EXT INPUT is set to ON and OSC IN is set to OFF.)

1.6 INSPECTING YOUR NEW INSTRUMENT

Inspect your new instrument for shipping damage when you receive it. If any damage is noted, immediately notify EG&G Instruments and file a claim with the carrier. Save the shipping container for possible inspection by the carrier.

Warning! *If your instrument has been damaged, its protective grounding may not work. Do not operate damaged equipment! Tag it to indicate to a potential user that it is unsafe to operate.*

1.7 MAINTENANCE AND SERVICE

Introduction

The Model 263A Potentiostat/Galvanostat has been designed for optimum reliability, and requires no periodic maintenance except for battery replacement.

This manual contains no service information, as there are no operator-serviceable parts inside the Model 263A. The instrument is very difficult to service in the field; special fixtures and services are required that are not readily obtainable except at the factory or at certain affiliate facilities. Contact the factory service department or the affiliate in your area if service is required.

Battery Replacement

After several years of use, the internal battery may require replacement. The Model 263A is equipped with a long-life lithium battery to retain the calibration settings in memory. The battery has a life of approximately two to five years, depending on how much you use the instrument. You cannot replace the battery yourself; you must send the instrument to an authorized EG&G PARC repair facility for battery replacement.

When the battery needs replacement, the instrument will alert you with a message on the front-panel LCD display (see Section 5.10). You can use the instrument without the battery, but you will have to perform the automatic calibration procedure every time you power up. This is, however, a simple procedure.

Cleaning Instructions

To clean the exterior of the instrument, proceed as follows.

1. Unplug the instrument.
2. Remove loose dirt on the outside of the instrument with a lint-free cloth.
3. Remove remaining dirt with a lint-free cloth dampened in a general purpose detergent and water solution. Do not use abrasive cleaners.
4. Allow sufficient time for the instruments to dry before reconnecting the power cord.

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Chapter 2
Safety Considerations

2.1 OVERVIEW

This instrument has been supplied in a safe condition. To ensure its continued safe operation, please read the following sections.

This chapter also includes information on possible radio frequency interference and transient sensitivity.

Note: The following symbol may appear beside certain connectors on the 263A. It indicates that additional safety information may be found in this manual. Refer to the appropriate section for the particular connector.



2.2 POWER CORD

For safe operation, the Model 263A must be electrically connected to earth ground through a suitable protective conductor. This connection is made via the earth ground prong of the Model 263A's power cord plug. Thus the power cord plug must be inserted into a socket outlet provided with the required earth ground contact. The protective action must not be defeated by the use of an extension cord without a protective conductor, by use of an "adapter" that doesn't maintain earth ground continuity, or by any other means.

Wherever possible, the instrument is furnished with a power cord plug that is compatible with the type of power socket in use where the instrument will be operated. If you have to provide a power cord, it should be an approved type with a standard IEC connector at one end for connection to the Model 263A.

Warning! *If it is necessary to replace the power cord or the power cord plug, the replacement cord or plug must have the same polarity as the original. Otherwise a safety hazard from electrical shock, which could result in personnel injury or death, might result.*

The wires in the power cord supplied by EG&G PARC are color-coded to denote polarity. Whatever the actual plug configuration, the black wire should be the line or active conductor (also called "live" or "hot"), the white wire should be neutral, and the green wire should be earth ground.

2.3 LINE VOLTAGE

Before plugging in the power cord, make sure that the Model 263A is set to the voltage of the ac power supply.

To confirm that the Model 263A is set for the correct line voltage: look at the Power Input module, located on the right side of the back panel. Note that there are four small line-voltage indicator windows near the lower edge of the module. These windows are aligned with the four available line-voltage settings on the cover module, 100V, 120V, 220V and 240V. The selected voltage is indicated by the color red showing through the corresponding window. For example, if the selected line voltage is 120V, you will see red through the 120V window. The other three will be black.

If the Model 263A is not set for the line voltage available at your site, refer to Chapter 3, Section 3.3, for instructions on how to reset it. Section 3.3 also describes how to replace the main power fuse.

2.4 DEFECTS AND ABNORMAL STRESSES

Warning! *If your instrument has been damaged, its protective grounding may not work. Do not operate damaged equipment! Tag it to indicate to a potential user that it is unsafe to operate.*

The ground protection is likely to be impaired if, for example, the instrument:

- Shows visible damage,
- Fails to perform the intended measurement,
- Has been subjected to prolonged storage under unfavorable conditions.
- Has been subjected to severe transport stresses.

The instrument should not be used until its safety has been verified by qualified service personnel.

2.5 VENTILATION

To maintain a safe operating temperature, it is necessary to allow some free space (minimum 10 cm) at the sides and rear of the instrument so that adequate air circulation can occur. Moreover, there must be adequate circulation between the spaces at the sides of the instrument and the general laboratory circulation to allow effective cooling. In a typical installation, these requirements are satisfied with a large safety margin. If the Model 263A is cabinet or rack mounted, some additional effort to assure adequate ventilation may be required. The ambient temperature should not exceed 45° C (113° F).

Note: Units having the 94 Option are equipped with a variable-speed fan mounted on the inside of the back panel. Temperature sensors control the fan as required to provide extra cooling in high-current operation. Take particular care that the rear air-circulation paths to the rear of the instrument and to the right side panel (as

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viewed from the front) are obstruction free in these units. *The ventilation input openings are on the right side. In units not having the 94 Option, the fan mounting hole is covered with a plate.*

2.6 RADIO FREQUENCY INTERFERENCE

In a typical application, it is unlikely that this instrument will act as a source of noticeable radio frequency interference. However, when operated near particularly sensitive equipment, interference emanating from this instrument could be a problem. Should this be the case, steps can be taken to minimize that interference. A discussion of the recommended steps follows.

Interference below about 10 MHz is most likely to be caused by radio-frequency currents flowing in the input and output cables, in the digital interface cables, or in the power line cord. The use of coaxial cables in making the analog signal input/output connection will usually prevent these lines from becoming a source of "below 10 MHz" radio frequency interference.

Two approaches are suggested for reducing interference that has its source in the digital interface (GPIB or RS-232) cables. The first is simply to shield these cables. The second is to provide a heavy ground connection between the grounds of all equipment sharing the interface bus. This is accomplished by strapping the chassis together with a braided or solid metal strap. (A solid strap does a better job but is more clumsy. Copper, aluminum, or brass are the recommended materials).

Because the Model 263A has an internal low-pass filter connected to the power line, the ac line cord is unlikely to be a source of radio frequency interference. If the internal filter seems to be inadequate, try decoupling the power line with an external filter. At frequencies below 100 kHz, an external isolation transformer could be helpful.

Warning!

To reduce the risk of potentially dangerous electrical shock, this work should only be performed by a qualified service technician, and then only with the instrument disconnected from all sources of power.

At frequencies above 10 MHz, these measures may not suffice to prevent radiation from being a problem, particularly at VHF frequencies. Additional measures will then be required. Shielding is generally effective. A suitable shield can be constructed using metal foil, wire screening, or similar materials.

Once the instrument is completely surrounded by the shield (taking care not to unduly restrict ventilation), the only additional requirement is to install low-pass filters where lines pass through the shield (all openings through the shield should be as small as possible). A capacitor between a line and the shield can function as a suitable low-pass filter. The leads of the capacitor should be as short as possible. This requirement is optimally satisfied by using coaxial feed-though capacitors.

In the case of a signal lead, it is essential that the capacitor's value be such as to attenuate the interference frequencies without unduly attenuating critical frequency components of the signal itself. The need to keep filter capacitor leads short cannot be overemphasized. Long leads establish sizable ground loops and may additionally act as radiating antennae.

Coaxial cables are a special case in that the cable shield acts as an extension of the enclosure shield. This being the case, the filter can be mounted in a shielded box fitted with coaxial connectors without undue concern for keeping the box extremely close to the enclosure. If more convenient to do so, it can be located at some distance from the enclosure as long as the integrity of the coaxial shield is maintained.

The techniques described are extraordinary measures that should be required for unusual cases only. If they are applied with care, radio frequency interference should be reduced to an acceptably low level in all but the most critical applications. However, if these techniques are applied incorrectly, the efforts to reduce the interference could prove disappointing. Users are advised to contact the factory for advice in the case of a problem that does not yield to these measures.

2.7 TRANSIENT SENSITIVITY

Generally speaking, the design and construction techniques used in equipment manufactured by EG&G PARC are conducive to assuring normal operation in the presence of moderate transient levels. Although these provisions are sufficient for operation in most places where this equipment is used, it is certainly possible for transient levels in particular environments to be so severe as to make reliable operation uncertain. High-level transients are of three general types.

1. **Static Discharge:** Transients from this source generally affect input or output circuits. Input circuits that include MOS field-effect transistors to achieve a high input impedance are particularly susceptible to damage from this source. Damage typically occurs when the charge built up on a user's body discharges into an input or output connector as a connection is being made. Among the factors determining the tendency for charges to build are the kind of clothing fabrics worn, shoe materials, and the materials in the floor or floor covering.

2. **High-Level Transients Generated Internal to the Place of Use:** Such transients almost always enter the instrument via the line cord. Possible sources include heavy-duty electric motors, rf equipment, lasers, diathermy machines, arc welders, spark chambers, and others.

3. **Lightning:** Unless the equipment is connected to remote sensors, or other devices so located as to be vulnerable to lightning strikes, transients caused by

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lightning almost always enter the instrument via the line cord.

Static discharge problems can sometimes be avoided by judiciously selecting the floor covering in the work area. The simplest approach to the problem is to discharge one's body by touching a grounded metal object just before touching the instrument, particularly when making connections to it. Transients that enter the instrument via the line cord can generally be suppressed by external line-transient filters. A suitable transient suppressor is available from EG&G Princeton Applied Research. Various kinds of power line conditioners are also commercially available.

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Chapter 3
Connecting Your System

3.1 INTRODUCTION

Nine connectors and an ac power/fuse assembly are located on the front and rear panels of the standard Model 263A. This chapter describes the functions of these connectors, and shows you how to connect your instrument to the test cell, pen recorder, and other equipment you may wish to use with it. It also shows how to set the instrument for the power line voltages available in different countries, and how to change the line fuse. Pinouts for the multi-pin connectors are provided in the appendix.

Before you begin to run your first experiment, you should perform the initial checks described in Chapter 4 to make sure your new instrument has not been damaged during shipping. The check-out procedure in Chapter 4 also will serve to introduce you its operation. Then, refer to Chapter 5 for instructions on how run your experiment.

3.2 FRONT PANEL CONNECTORS (Figure 3-1)

EXT INPUT Connector: This BNC connector has two separate but similar functions: External Input and Oscillator Input. The main difference between these functions is that in the OSC IN mode the gain of the applied signal can be scaled up or down, while the EXT INPUT mode always applies unity gain to the input signal.

This connector can be made active (turned on) or inactive either with the front panel controls or by software commands from an external computer. From the front panel, it can be controlled with either the EXT INPUT or the OSC IN setting on the [System Default] menu (see "Adding an External Signal" in Section 3.4). Remote control from an attached computer is described in the *Model 263A Command Set Handbook*. Note that both EXT INPUT and OSC IN cannot be set to ON at the same time.

When this connector is active ("ON"), an analog control voltage applied to it will be summed with the output of the digital-to-analog converters (DACs), which are set at the front panel or from an attached PC. The control signals could be generated by, for example, an EG&G PARC Model 175 Universal Programmer to provide rapid scanning and true analog cyclic voltammetry. Other waveform generators, a lock-in amplifier, or a frequency response analyzer could be used.

In potentiostatic operation, all of the control potentials add algebraically. For example, if the voltage applied to EXT INPUT is +0.5 V, at a time when a potential of +0.5 V is programmed in the instrument, the net potential applied will be +1.0 V, and the working electrode would be controlled at +1.0 V with respect to the reference electrode. (This assumes, of course, that the applied signal is not scaled by selecting the OSC IN mode.) A positive applied potential will make the current tend to be more anodic. A negative applied potential will make the current tend to be more cathodic.

In galvanostatic operation, making the applied potential more positive by any means except applying a potential to the EXT INPUT connector will tend to make the cell current more cathodic. Making the applied potential more negative will tend to make the current more anodic. *This sense is reversed at the EXT INPUT connector. There, making the input more positive will make the cell current more anodic. Making the input more negative will make the cell current more cathodic.*

In potentiostat operation, the maximum voltage that can be safely applied to the connector is ±10 V in the EXT INPUT mode and ±5 V in the OSC IN mode. In galvanostat operation, the safe range in either mode is -2 V to +2 V (corresponding to ±2 times the full-scale current). Input impedance at the connector is 100 kΩ.

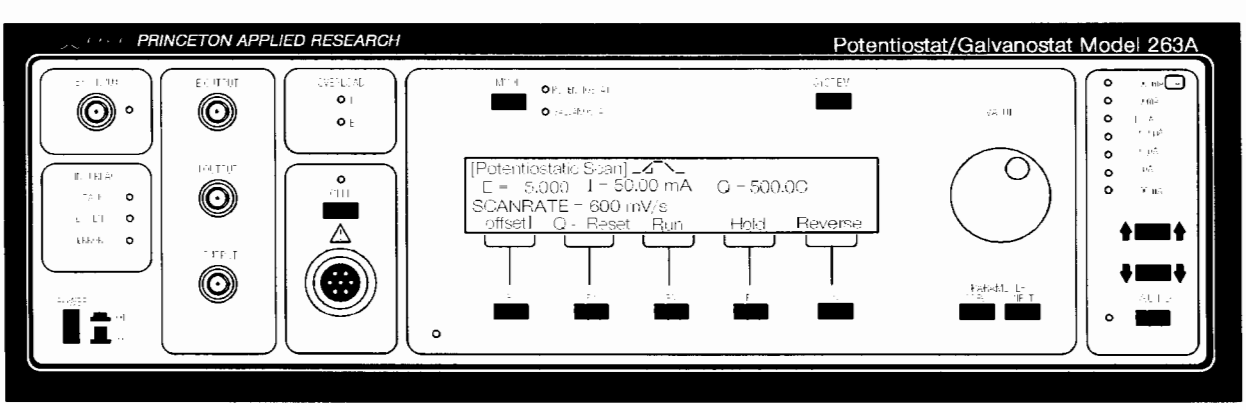


Figure 3-1. FRONT PANEL OF MODEL 263A

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E OUTPUT Connector: This dc-coupled BNC connector can be used to monitor the polarity and amplitude of the working electrode potential versus the reference electrode in the test cell. It can be used to drive the X-axis of a pen recorder or oscilloscope. If positive feedback or current interrupt iR compensation is enabled (Chapter 6), the potential at E OUTPUT is corrected for uncompensated resistance (R_U). ***Note:*** *In units equipped with the 98 or 99 Option, up to ±10 V of suppression, set by the EOUTSUP command (see discussion of this command in Command Set Handbook), can be provided at this output. Figures A-4 and A-5 in Appendix A, which illustrate the 98 and 99 Options respectively, show the suppression DAC connections.*

I OUTPUT Connector: The potential at this dc-coupled connector represents the ratio of cell current to full-scale current at the current range selected, with 1 volt representing unity or 100% of full-scale current.

For example, with the 10 μA full-scale range selected, 0.5 V at I OUTPUT indicates that cell current is one half of full scale, or 5.0 μA. Or, if the 100 μA range is selected, 1.25 V at I OUTPUT would indicate that cell current is 125% of full scale, or 125 μA. Currents as high as 2 × full scale can be accommodated.

The polarity of the potential at the I OUTPUT connector is the same as the polarity of the cell current. On the 1 mA current range, for example, a current level of -1 mA will give -1 V at the I OUTPUT connector, representing an anodic or reducing current at the working electrode. This connector can be used to drive the Y-axis of a pen recorder or oscilloscope.

Note: *In units equipped with the 98 or 99 Option, suppresion up to ±4 times the selected current range can be provided at this output. The supression level is set by the IOUTSUP command (see discussion of this command in Command Set Handbook), Figures A-4 and A-5 in Appendix A, which illustrate the 98 and 99 Options respectively, show the suppression DAC connections.*

OUTPUT Connector: When the Model 263A is operated remotely from an external computer, this ac-coupled connector can be used to monitor either cell current or cell voltage (as determined by the MIE command). The default is current. The OUTPUT connector cannot be controlled from the front panel of the Model 263A. Thus, under local control, the connector can monitor current only. This connector is used mainly with EG&G PARC electrochemical impedance systems.

When the instrument is monitoring cell current, the potential at this connector is the same as that at the I OUTPUT connector, except that any dc component of the signal is blocked by the ac coupling. As with the I OUTPUT connector, the potential represents the ratio of test cell current to full-scale current at the current range selected, with 1 volt representing unity or 100% of full-scale current. Again, currents as high as 2 × full scale can be accommodated.

When the instrument is monitoring cell potential, the signal at this connector is the same as that at the E OUTPUT connector, except that any dc component of the signal is blocked by the ac coupling. The connector is used to monitor the polarity and amplitude of the working electrode potential versus the reference electrode in the test cell. The output range is ±10 V. If positive feedback or current interrupt iR compensation is enabled, the potential at the connector is corrected for uncompensated resistance (R_U).



CELL Connector: The cell cable connects to the 7-pin CELL connector on the front panel. The cell cable has a 7-pin LEMO connector that is rugged and reliable, and will not become disconnected inadvertently. The LEMO connector is keyed so it it can only be inserted the correct way. To connect it, grasp it by the grooved sleeve, turn it so the red dot aligns with the red mark on the CELL connector (this will ensure that the key aligns with the keyway), and press it into place.

To disconnect the cell connector, grasp the grooved sleeve and pull straight out.

At the other end of the cable are four color-coded leads, three of which terminate in alligator clips and the fourth in a pin-jack socket. In both potentiostatic and galvanostatic operation, the red clip connects to the counter electrode and the green clip connects to the working electrode. The reference electrode plugs directly into the pin-jack socket (white) and the black clip is ground.

Use of the black lead depends on a number of factors. It is not ordinarily used with the Model 263A, although it is available if needed for a special purpose, such as supplying ground to a shield-screen surrounding the cell.

An exception is in making galvanic corrosion measurements, in which the black lead connects to one of the metal working electrodes and the green lead connects to the other. The reference electrode (white) is connected in the usual manner and the red (counter-electrode) lead is left unconnected.

Potentials as high as ±20 V at currents as high as 200 mA (2 A with 94 option) may be present at the counter electrode lead of the cell cable. **The high current capability requires that reasonable care be taken in handling these leads**. The CELL switch should always be off when making the cell connections. *Don't rely on the indicator*. It should similarly be off when the cable leads are being examined or disconnected.

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Voltage Levels on Systems Equipped with /99 Option: A unit operated in Float mode (set via the rear-panel FLOAT-NORMAL switch) may have potentials of up to ±300 V on any or all pins of the Cell connector, depending on the nature and configuration of the cell to which the unit is connected. Extreme caution should be exercised when inserting or removing these connectors in a floating system.

3.3 REAR PANEL CONNECTORS (Figure 3-2)

POWER INPUT: Wherever possible, the instrument is furnished with a power cord plug that is compatible with the type of power socket in use where the instrument will be operated. If you have to provide a power cord, it should be an approved type with a standard IEC connector at one end for connection to the Model 263A.

Warning! *If it is necessary to replace the power cord or the power cord plug, the replacement cord or plug must have the same polarity as the original. Otherwise a safety hazard from electrical shock, which could result in personnel injury or death, might result.*

The wires in the power cord supplied by EG&G PARC are color-coded to denote polarity. Whatever the actual plug configuration, the black wire should be the line or active conductor (also called "live" or "hot"), the white wire should be neutral, and the green wire should be earth ground.

Before connecting the power cord to the power source, make sure that the Model 263A is set for the voltage of the available ac supply.

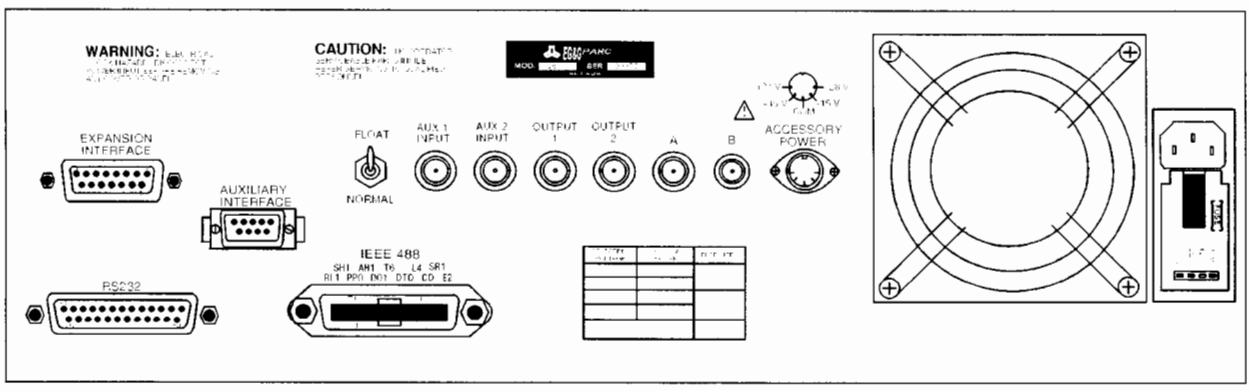


Figure 3-2. REAR PANEL OF MODEL 263A

LINE VOLTAGESELECTION: Referring to Figure 3-3, note that there are four small line-voltage indicator qwindows near the lower edge of the module. These windows are aligned with the four available line-voltage settings on the cover module, 100V, 120V, 220V and 240V. The selected voltage is indicated by the color red showing through the corresponding window. For example, if the selected line voltage is 120V, you will see red through the 120V window. The other three will be black.

The color you see through any window is determined by the position Of the internal line-voltage selector card, which can be installed in any of four different orientations, each corresponding to a different line voltage. The procedure for selecting the line voltage follows.

1. Disconnect the power cord from the 263A.

2. Open the Power Module door by pressing at the point indicated in the left-hand view of Figure 3-3. Because the door is hinged directly beneath the point where pressure is applied, you will need to push inwards, but with some upward thrust so that the door will swing open, exposing the interior of the module as shown in the righthand view of Figure 3-3.

3. Using needle-nose pliers, grasp the Voltage Selector Card firmly and pull it straight out of the module. Note that each edge of the card has the red dot in a different location, each corresponding to a different line voltage configuration selection.

4. Orient the card so that the red dot corresponding to the desired line voltage selection will align with the line voltage indication on the cover after the cover is closed. Then install the card by pressing it firmly into its socket.

5. If necessary, change the fuse to the type appropriate for the new line-voltage selection.

6. Close the cover. The red dot should appear in the window corresponding to the desired line-voltage configuration.

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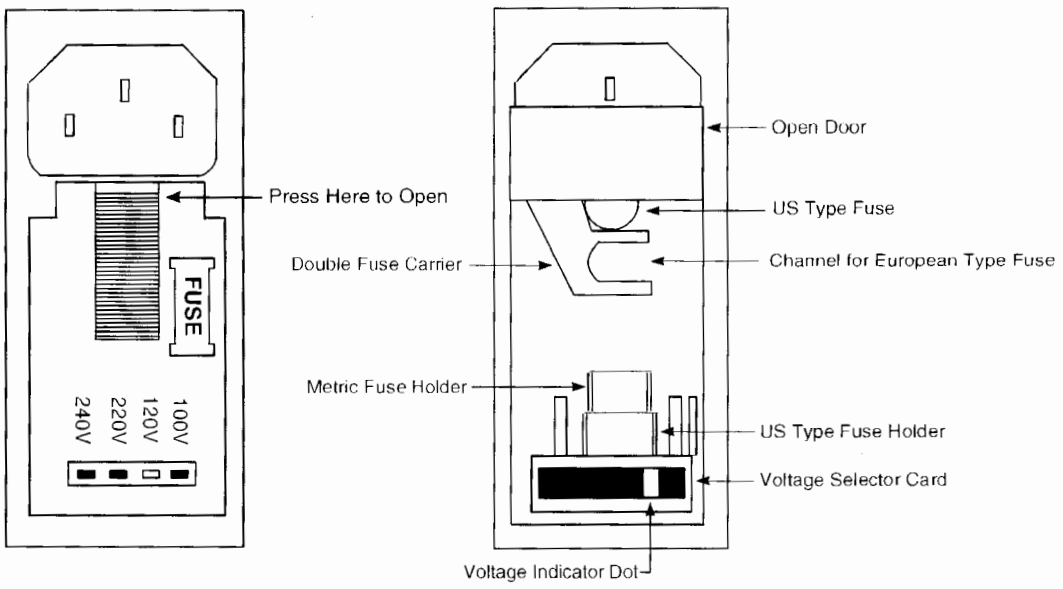


Figure 3-3. POWER INPUT MODULE.

7. Reconnect the power cord to the 263A.

FUSE REPLACEMENT: The Power Input module has a double fuse carrier which can accept either a US type fuse or a European type metric fuse. This carrier is located on the inner surface of the module door and becomes accessible when the door is opened. Only one fuse can be installed at any time. The required fuse rating is printed on the panel near the Power Input module.

To replace the fuse:

1. Disconnect the power cord from the 263A.

2. Open the Power Module door by pressing at the point indicated in the left-hand view of Figure 3-3. Because the door is hinged directly beneath the point where pressure is applied, you will need to push in but with some upward thrust so that the door will swing open, exposing the interior of the module as shown in the righthand view of Figure 3. When the door opens, the fuse comes free of the fuse holder and remains in the fuse carrier.

3. Remove the defective fuse and install the replacement fuse in its place. No particular precision is required in placing the fuse in its channel. The fuse will be automatically positioned correctly in the fuse holder when the door is closed. Be sure to use the correct channel of the fuse carrier. Figure 3-3 shows a US type fuse installed.

4. Close the door. As it closes the fuse will be correctly positioned and pressed into the proper fuse holder.

5. Reconnect the power cord.



ACCESSORY POWER: Provides ±26 V regulated and ±15 V regulated power for accessory equipment. Up to about 50 mA may be safely drawn from each of these sources. The voltage available at each connector pin is defined in the appendix.

Voltage Levels on Systems Equipped with /99 Option: A unit operated in Float mode (set via the rear-panel FLOAT-NORMAL switch) may have potentials of up to ±300 V on any or all pins of the Accessory Power connector, depending on the nature and configuration of the cell to which the unit is connected. Extreme caution should be exercised when inserting or removing these connectors in a floating system.

AUXILIARYINTERFACE: When the Model 263A is controlled from an external computer, this connector, via the Model 507 Interface, can provide control signals for a Model 303A Static Mercury Drop Electrode or Model 616 Rotating Electrode System.

It can send Purge, Stir, and Dispense signals to the Model 303A. But note that the Model 303A is not cabled directly to this connector. It is cabled to the Model 507 Interface unit, which in turn is cabled to the Model 263A AUXILIARY INTERFACE connector.

The Model 263A can also turn the Model 616 Rotating Electrode System on and off with the STIR signal. In this case the Model 616 is cabled directly to this connector.

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RS-232 Connector: Serial port for control of the Model 263A from an external computer via an RS-232 interface cable. The communications parameters are set from the front panel of the Model 263A. **Note:** The maximum potential that can be applied to this port is ±15 V. The *Model 263A Command Set Handbook* describes communications protocols and settings. See the appendix for a pinout listing.

IEEE-488 Connector: Parallel port for control of the Model 263A from an external computer via an IEEE-488 (GPIB) interface cable. The GPIB address, terminator characters, and status of the Test Echo function are set from the front panel of the Model 263A. The *Model 263A Command Set Handbook* describes communications protocols and settings. See the appendix for a pinout listing.

3.4 HOW TO CONNECT YOUR SYSTEM

This section tells you how to connect the Model 263A to the corrosion cell, and to a computer and other equipment you may wish to use.

Siting and Ventilation

Before setting up and connecting your instrument, give some thought to finding a safe and convenient location for it. If desired, it may be mounted in a standard 19-in. (47.5 cm) equipment rack assembly. Instructions for rack mounting are provided in the following section.

To maintain a safe operating temperature, it is necessary to allow some free space (minimum 10 cm) at the rear and at both sides of the instrument so that adequate air circulation can occur. Moreover, there must be adequate circulation between the spaces at the sides of the instrument and the air circulation in your laboratory to allow effective cooling. In a typical installation, these requirements are satisfied with a large safety margin. If the Model 263A is cabinet or rack mounted, some additional effort to assure adequate ventilation may be required. In particular, if the unit is equipped with the 94 Option it is critical that there be good air flow to the ventilation intake openings in the right side panel (as viewed from the front). The ambient temperature should not exceed 45° C (113° F).

Rack Mounting

A kit for installing the Model 263A in a standard 19-in. (47.5 cm) equipment rack is available from EG&G PARC (Accessory Kit K0288). It contains a pair of mounting brackets that must be installed on the instrument to adapt it for rack mounting.

To install the mounting brackets, use a 3-mm hex key or hex driver to remove the socket-head screws that secure the handles and the adapter blocks behind them. Put the adapter blocks aside. Then, reuse the screws to mount the rack-mounting brackets between the Model 263A case and the handles.

After you have attached the mounting ears, slide the instrument into the rack and secure it with the hardware provided by the rack supplier. Then, connect the cables as described in this chapter. Be sure to follow the ventilation recommendations discussed in the previous section.



Connecting the Cell

To connect the cell cable to the Model 263A:

1. Grasp the connector on the cell cable by the grooved sleeve, turn it so the red dot aligns with the red mark on the CELL connector, and press it into place.

   (To disconnect the cell cable connector, grasp the grooved sleeve and pull straight out.)

2. Assemble the electrochemical test cell and prepare it with the test solution.

3. Connect the color-coded leads on the cell cable to the electrodes in the test cell. There are four leads, three of which terminate in alligator clips and the fourth in a pin-jack socket.

   Warning!
   To prevent possibly dangerous electric shock, always have the front-panel CELL switch off when handling the clip leads at the end of the cell cable. To assure your safety as well as that of the equipment, get in the habit of always checking that the panel Cell LED is *not* ON.

   In both potentiostatic and galvanostatic operation (except for galvanic corrosion measurements—see below), the red clip connects to the counter electrode and the green clip connects to the working electrode. The reference electrode plugs directly into the pin-jack socket (white) and the black clip is ground.

   The polarity of the potential at the counter electrode will be opposite the "applied potential." This is necessary to establish the correct polarity relationship at the working electrode versus the reference electrode.

   Use of the black lead depends on a number of factors. In most cases it is not used, although it is available if needed for a special purpose such as providing ground to a shield or screen surrounding the cell.

   An exception is in making galvanic corrosion measurements. In this technique, the black lead connects to one of the metal working electrodes and the green lead connects to the other working electrode. The reference electrode (white) is connected in the usual manner and the red (counter electrode) lead is left unconnected.

   Take care that the lead clips do not short together. Because the ground lead is often unused, it tends to

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be overlooked, making the possibility of an accidental short involving this lead more of a problem than for the other two.

4. If the Model 263A is not set to the correct mode for your experiment, press the MODE switch as many times as necessary to display the menu screen for the mode you wish to use. Refer to Chapter 5 for operating instructions.

5. When you are ready to start running your experiment, press the CELL switch to on.

Remote Control From a Host Computer

The Model 263A can be controlled remotely from a personal computer running standard EG&G PARC electrochemistry software. Instructions for remote operation using the EG&G PARC Electrochemical Command Set built into the Model 263A are given in the separately bound *Model 263A Command Set Handbook*.

Either the RS-232 serial port or the IEEE-488 (GPIB) port on the rear panel of the Model 263A can be used for communications with the host computer. If the IEEE-488 interface is used, the host computer must be equipped with a GPIB interface card. For details of hardware requirements, refer to the manual for the software to be run on the host computer. EG&G PARC software is designed to interface an IBM or IBM-compatible computer equipped with a National Instruments PC2, PC-2A, or PC-AT (non-Microchannel) GPIB card or with a National Instruments MC-GPIB (Microchannel) GPIB card.

Before the Model 263A and a host computer can successfully talk to each other, certain communications parameters have to be set in both. For GPIB communications, the instrument must be set to the address assigned to it at the computer, and the terminator character required by the host must be set. For RS-232 communications, the baud rate, parity, number of data and stop bits, and terminator character must match the host settings.

Note that with most software running on an IBM-compatible computer, the terminator must be a CR and both serial and GPIB echo must be off. Further details of communications protocols for GPIB and RS-232 interfacing are given in Appendixes A, B, and C of the *Model 263A Command Set Handbook*.

To set the communications parameters on the Model 263A, move to the [System Interface] menu on the LCD panel by pressing the SYSTEM key. Then, press the PREV or NEXT PARAMETER key until a parameter to be set appears on the third line of the display. Turn the VALUE knob to set the parameter. Press the PREV or NEXT PARAMETER key again to display the next communications parameter, and set it in the same fashion. Continue this process until all required parameters have been set. If you need more information on setting parameters, refer to Chapter 5.

Connecting a Pen Recorder or Oscilloscope

The potential of the working electrode with respect to the reference electrode is continuously provided at the E OUTPUT connector. This signal can be used to drive the X-axis drive signal line of a pen recorder or oscilloscope.

A meaningful E OUTPUT signal is provided as long as there is a reference electrode. If the reference electrode is not connected, as might be the case in galvanostatic operation, the E OUTPUT signal is undefined.

In Potentiostatic operation, if positive feedback or current interrupt iR Compensation is on, the compensation corrects the E OUTPUT signal level for the iR drop.

The E OUTPUT signal is analog and quite clean, free of the steps, spikes, and similar interference common to digitally produced signals. It is inverted with respect to the applied potential. For example, if the applied potential is +1 V, the potential at the E OUTPUT connector will be -1 V.

The output impedance at the E OUTPUT and I OUTPUT connectors is 1 kΩ. We suggest using either an EG&G PARC RE0150 X-Y recorder or an RE0151 X-Y-T recorder, as both instruments are designed for use with the Model 263A. Simply connect the X-axis drive signal line of the recorder to the E OUTPUT connector, and the Y-axis drive signal line to the I OUTPUT connector.

Adding an External Signal

The EXT INPUT connector on the front panel provides a means for summing external control potentials with those generated in the instrument or transmitted to it from an external computer. This gives you a way to add any customized waveform function you wish. The control signals could be generated by, for example, an EG&G PARC Model 175 Universal Programmer to provide rapid scanning and true analog cyclic voltammetry. Other waveform generators, a lock-in amplifier, or a frequency response analyzer could be used. The applied signal can be scaled up or down, if desired, by a factor of 2, 0.2, or 0.02.

To control the EXT INPUT connector from the front panel, press the SYSTEM key until the [System Default] menu appears on the LCD panel. If the applied signal has a suitable amplitude and you do not wish to scale it, press the PREV or NEXT PARAMETER key until "EXT INPUT =" appears on the third line of the menu. Then, turn the VALUE knob until "EXT INPUT = ON" is displayed. To disconnect, turn the knob until "EXT INPUT = OFF."

If the applied signal is too large or too small and you wish to scale it, press the NEXT PARAMETER key until "OSC IN =" is displayed on the third line of the menu. Turn the VALUE knob to "OSC IN = ON." Then, press the NEXT PARAMETER key again until "OSC GAIN =" appears on the third line of the [System Default] menu. Turn the VALUE knob to set the OSC GAIN factor to one of the three available values: 0.02, 0.2, or 2. To

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disconnect, set "OSC IN = OFF."

Note that both EXT INPUT and OSC IN cannot be set to ON at the same time. If you are unsuccessful in turning one on, check that the other is off.

When the connector is ON (connected), the EXT INPUT indicator lights and the instrument becomes responsive to potentials applied to the connector. When it is OFF (disconnected), the indicator goes out, indicating that potentials applied to this input will be ignored. Detailed instructions for using the LCD menu screen and associated controls are given in Chapter 5.

Connecting a Model 303A SMDE
When the Model 263A is controlled from an external computer, it can send PURGE, STIR, and DISPENSE signals to an EG&G PARC Model 303A Static Mercury Drop Electrode. Instructions for sending these commands are given in the *Model 263A Command Set Handbook*.

Also required is an EG&G PARC Model 507 Interface, which must be purchased separately. To control the Model 303A, connect it to the Model 507 Interface and the Interface unit to the AUXILIARY INTERFACE connector on the back of the Model 263A. The required cables are provided with the Model 507 Interface. Refer to the *Model 507 Interface Installation Guide*, EG&G PARC Part Number 222556, if you need more detailed installation instructions.

Connecting a Model 616 Rotating Disk Electrode
When the Model 263A is controlled from an external computer, it can send the STIR signal to an EG&G PARC Model 616 Rotating Disk Electrode (RDE) to turn it on and off. Instructions for sending the STIR command are given in the *Model 263A Command Set Handbook*. Connect the INPUT jack on the Model 616 to pin 9 of the AUXILIARY INTERFACE connector on the rear panel of the Model 263A.

Supplying Accessory Power
The ACCESSORY POWER connector on the rear panel can provide ±26 V regulated and ±15 V regulated power for accessory equipment. Up to about 50 mA may be safely drawn from each of these sources. The voltage available at each connector pin is defined in the appendix.



Voltage Levels on Systems Equipped with /99 Option:
A unit operated in Float mode (set via the rear-panel FLOAT-NORMAL switch) may have potentials of up to ±300 V on any or all pins of the Accessory Power connector, depending on the nature and configuration of the cell to which the unit is connected. Extreme caution should be exercised when inserting or removing these connectors in a floating system.

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Chapter 4
Initial Checks

4.1 INTRODUCTION

The sample session that follows can be used to check the performance of your new instrument and make sure it sustained no internal damage during shipping. If you have not used a Model 263A before, it also will help acquaint you with the operation of the instrument.

Before you set up your new instrument and apply power to it, inspect it for external shipping damage. Any damage noted should be reported to the carrier and to EG&G Princeton Applied Research. Be sure to save the shipping container for inspection by the carrier.

Warning! *If you have received a damaged instrument, its protective grounding may not work. Do not operate damaged equipment! Tag it to indicate to a potential user that it is unsafe to operate.*

This sample session uses an internal 10 kΩ resistor to provide a "dummy" load, substituting for a connected cell. No special test equipment is required. The checks are adequate for determining that the instrument has arrived in good working order and is functioning normally. They are not intended to demonstrate that the instrument meets specifications. Each Model 263A receives a careful checkout before leaving the factory and, if no shipping damage has occurred, will perform within the limits of the specifications.

If any problems are encountered in carrying out these checks, contact the factory or the factory-authorized representative in your area for aid.

4.2 POWER

Before plugging in the power cord, make sure that the Model 263A is set to the voltage of the available ac supply. Also, confirm that the power cord plug is the correct type (see Chapter 3, Section 3.3).

To confirm that the Model 263A is set for the correct line voltage: look at the Power Input module, located on the right side of the back panel. Note that there are four small line-voltage indicator windows near the lower edge of the module. These windows are aligned with the four available line-voltage settings on the cover module, 100V, 120V, 220V and 240V. The selected voltage is indicated by the color red showing through the corresponding window. For example, if the selected line voltage is 120V, you will see red through the 120V window. The other three will be black.

If the Model 263A is not set for the line voltage available at your site, refer to Chapter 3, Section 3.3, for instructions on how to reset it.

4.3 SETUP

1. Set the Model 263A on a lab bench in a convenient location. Or, if you prefer, mount it in a standard 19-in. (47.5 cm) rack assembly. Instructions for rack mounting are provided in Chapter 3, Section 3.4.

2. Connect the Model 263A to the ac power source.

3. Press the Model 263A's POWER switch to turn on the unit. Various indicator lamps on the front panel will light.

4. Make sure the CELL indicator on the front panel is not lit. The CELL indicator is located just above the CELL pushbutton. If the indicator is lit, press the CELL pushbutton once to extinguish it.

5. Connect the cell cable to the CELL connector on the front panel of the Model 263A. Grasp it by the grooved sleeve, turn it so the red marks align, and press it into place.

6. Press the SYSTEM key until the [System Default] menu is displayed on the LCD display. The SYSTEM key is located above the LCD panel.

7. Press the PREV or NEXT PARAMETER key until "CELL TYPE =" appears on the third line of the LCD display. These keys are located below the VALUE knob. Then, turn the VALUE knob until CELL TYPE = DUMMY is displayed. (In this setting the internal 10 kΩ dummy resistor acts as the cell.)

4.4 A SAMPLE SESSION

The following steps program the Model 263A to do a controlled-potential experiment on the internal 10 kΩ dummy cell resistor. A scan starting at 0 V, advancing to 1 V, and then returning to 0 V will be applied. The current in the resistor will track this voltage, starting at zero, increasing in the negative direction to -100 μA, and then decreasing to zero again. A voltage corresponding to this current will appear at both the I OUTPUT connector and at the OUTPUT connector. The current through the resistor and the voltage across it will be displayed throughout the experiment on the LCD panel.

Note: Analog components cannot be made to exhibit exactly their rated values, especially under varying temperature conditions. Thus analog components have tolerance ratings that specify acceptable ranges above and below their nominal values. The 10 kΩ dummy cell resistor, for example, has a very tight 0.01% tolerance, which means that its actual resistance in the circuit could be anywhere between 9,999 and 10,001 Ω. Also, residual drift or noise

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in the Model 263A itself can contribute a small additional deviation in both the current and potential indications. As a result of these uncertainty factors, the current indications on the LCD panel may be as much as 1% off with respect to the values indicated in this procedure.

1. Press the MODE key until the [Potentiostatic Scan] menu is displayed on the LCD display. The MODE key is above the LCD panel. The POTENTIOSTAT indicator should be lit, indicating that the Model 263A is in the controlled-potential mode.

2. Press the PREV or NEXT PARAMETER key until E INIT = appears on the third line of the LCD display. Then, turn the VALUE knob until E INIT = 0.000V is displayed. This enters 0 V as the programmed starting potential for the scan to be performed.

   As you would expect, the faster you turn the VALUE knob the faster the parameter value changes. But note that the relationship between knob speed and value change does not remain linear. Above a certain knob speed the processor "shifts gears" and changes to a higher ratio of parameter change to knob speed. This allows you to make large changes in the value of a parameter by spinning the knob rapidly.

3. Press the NEXT PARAMETER key once. "E MID =" should appear on the third line of the LCD display. Then, turn the VALUE knob until E MID = 1.000V is displayed. This establishes 1.000 V as the scan vertex potential.

4. Press the NEXT PARAMETER key again. "E FINAL =" should appear on the third line of the LCD display. Then, turn the VALUE knob until E FINAL = 0.000V is displayed. This establishes the scan-end potential at 0 V (which was also the starting potential).

5. Press the NEXT PARAMETER key again. "T INIT =" should appear on the third line of the LCD display. Then, turn the VALUE knob until T INIT = 10.0s is displayed. This enters 10 s as the time for which E INIT (0 V) will be applied before beginning the scan.

6. Press the NEXT PARAMETER key again. "T MID =" should appear on the third line of the LCD display. Then, turn the VALUE knob until T MID = 10.0s is displayed. This establishes 10 seconds as the time for which the mid or vertex potential (E MID) will be applied.

7. Press the NEXT PARAMETER key again. "T FINAL =" should appear on the third line of the LCD display. Then, turn the VALUE knob until T FINAL = 10.0s is displayed. This establishes 10 seconds as the time for which the scan-end potential (E FINAL) will be applied.

8. Press the NEXT PARAMETER key again. "SCAN RATE =" should appear on the third line of the LCD display. Then, turn the VALUE knob until SCAN RATE = 20.0 mV/s is displayed. (The scan rate is the rate of change of the applied potential.)

9. Press the NEXT PARAMETER key again. "CYCLES =" should appear on the third line of the LCD display. Then, turn the VALUE knob until CYCLES = FULL is displayed. This programs the Model 263A to scan the potential applied at the working electrode between E INIT, E MID, and E FINAL for one complete cycle, with the scan rate and time delays you have specified, and then end the experiment. FULL CYCLE is defined in Chapter 8 under Cycle Control.

10. Press the NEXT PARAMETER key again. "CELL @ END =" should appear on the third line of the LCD display. Then, turn the VALUE knob until CELL @ END = E FINAL is displayed. This will cause the cell to remain at the scan-end potential (E FINAL) after the scan is complete.

11. The automatic full-scale current range setting mode must be turned off for this test. Make sure the AUTO indicator at the lower right side of the front panel is not lit. If it is, press the AUTO pushbutton to turn off the automatic mode and enable manual setting.

12. Press the [↑ ↑] or [↓ ↓] key above the AUTO pushbutton as necessary to light the 100 μA full-scale current range indicator.

13. Press the CELL switch to enable the cell connector. The CELL indicator just above it should light, showing that the cell is connected.

    The Model 263A is now ready to run the experiment, using its internal dummy cell resistor as a substitute for an actual cell. The programmed sequence will be initiated in the following step.

14. Press "Start". (Note that on this menu the F3 key now is assigned the "Start" function.) The following should be observed.

    a. F3 has been reassigned as "Stop". You can press F3 to stop your experiment at any time. (F3 will be assigned the "Start" function again, and you may press it at any time to restart the checkout sequence.)

    b. The initial potential (0 V) and the resulting current (0 μA) are indicated on the second line of the LCD display.

    Note: As mentioned before, there is always some

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small deviation from the nominal value. For purposes of these initial checks, a normal current indication will be within 1% of the selected current range (range is 100 μA; 1% of that is 1 μA). Potential indications should be within 10 mV of the indicated value.

c. After the programmed initial delay interval (T INIT = 10.0s), the scan will begin and the displayed potential and current will begin to increase. The potential will start at 0 V (E INIT) and increase gradually to 1 V (E MID). It will remain at 1 V for 10 seconds (T MID). Then, it will gradually decrease to 0 V (E FINAL). It will remain at 0 V for 10 more seconds (T FINAL). The current will track this voltage, starting at zero, increasing in the negative direction to -100 μA, and then decreasing to zero again.

15. Press the CELL key once to disconnect the cell. The associated indicator light should extinguish.

16. Press the Model 263A POWER switch to turn off power to the unit. The indicator lights will extinguish and the LCD panel will blank.

This completes the initial checks. If the Model 263A behaved as indicated above, you can be reasonably confident that all its circuits are functioning normally.

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Chapter 5
Getting Started

5.1 INTRODUCTION

Although the Model 263A is a complex and sophisticated instrument, it is easy to use. It can be controlled either directly from its front panel or from a personal computer.

This chapter describes the functions of the controls, LCD display, and indicator lights on the front panel. It then shows you how to set up the basic experiment parameters that are common to both potentiostatic and galvanostatic operation from the front panel. These parameters seldom require changing. It also shows how to set the RS-232 or GPIB (IEEE-488) communications parameters necessary for controlling the instrument from a host computer.

After you have set these parameters, you must also set the parameters that are specific to the kind of experiment you wish to do. To complete your preparation and run your experiment, continue to either

- Chapter 6 to set potentiostatic parameters and run a potentiostatic experiment, or

- Chapter 7 to set galvanostatic parameters and run a galvanostatic experiment.

A battery-powered parameter backup system retains all selections and parameter values. When the Model 263A is powered up, all selections and parameter values are as they were at the end of the previous operating session. If you wish to restore the default values, hold in any of the keys on the front panel as you power up.

Connector functions, and instructions for connecting your instrument to the test cell, pen recorder, and other equipment you may wish to run with it, are given in Chapter 3. Instructions for operating the unit remotely via either the RS-232 or GPIB interface port are provided in the separately bound *Model 263A Command Set Handbook*.

Note: Many of the parameters set from a remote computer via the GPIB or RS-232 bus are stored in memory locations shared with similar parameters set from the front panel. In some circumstances this can cause unpredictable interactions between remote parameters and front-panel parameters. We recommend that you do not mix front-panel and remote operations.

Certain conditions that interfere with normal operation will cause error messages to be displayed on the LCD panel. These messages are explained in Section 5.10.

5.2 OPERATING MODES

This instrument can operate in either of two basic operating modes, Potentiostat or Galvanostat. The mode in which it is operating at any time is indicated by an illuminated lamp.

In each of these basic operating modes, the cell potential or current can be held constant to extremely close tolerances, or it can be varied in closely controlled increments. These operating modes are explained below. To switch between modes, simply press the MODE key on the front panel.

Potentiostatic

In this mode, the Model 263A controls the potential at the working electrode with respect to the reference electrode. The potential at the counter electrode is driven to the potential required (consistent with the ± 20 V compliance of the control amplifier) to establish the desired working electrode potential. The range over which the working electrode potential can be controlled is ± 10 V, although only a ± 2 V range can be used in a single experiment. *This range is increased to ± 8 V with the 91 option.*

Galvanostatic

In this mode, the Model 263A controls the current between the counter and working electrodes at the specified fraction of the selected current range (up to the maximum of two times the current range). The counter electrode is driven to the potential required (consistent with the ± 20 V compliance of the control amplifier) to establish the desired cell current. The reference electrode is not used in the control loop, but can be used to monitor the potential at some point in the electrochemical cell.

Scanning and Stepping Transitions

Under front-panel control, the Model 263A can hold the cell potential or current fixed at a single level during the experiment, or it can vary the potential or current between three separate programmable levels. In potentiostatic operation, the separate levels are called E Initial, E Mid, and E Final. In Galvanostatic operation, the levels are called I Initial, I Mid, and I Final.

The transition between the three levels can be in the form of either a gradual ramp or "scan" between levels, or a sudden sharp "step." So there are actually a total of six modes of operation available:

- Potentiostatic, where cell potential is held constant.

- Potentiostatic Scan, with a gradual ramp between the three levels of cell potential. You can program the rate of change ("SCANRATE") of the potential between levels.

- Potentiostatic Step, with sharp steps between the three levels of cell potential.

- Galvanostatic, where cell current is held constant.

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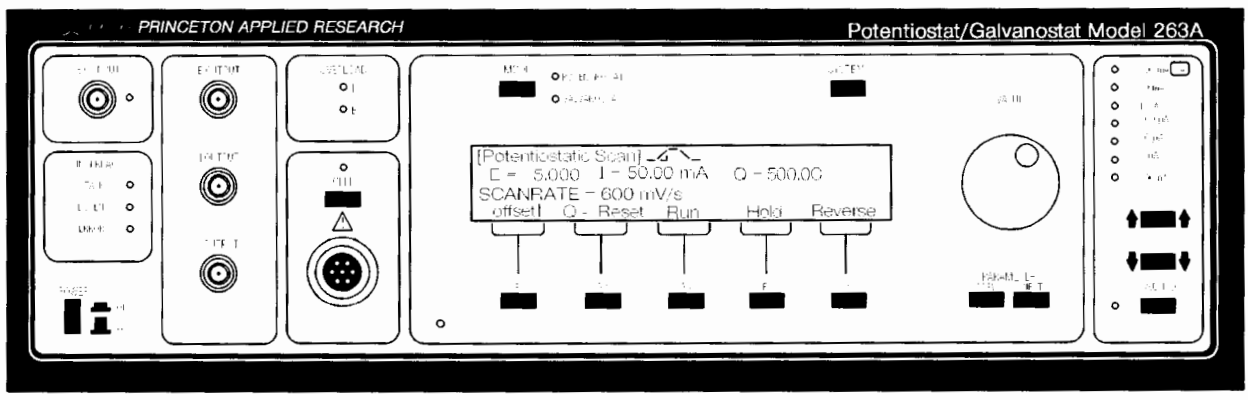


Figure 5-1. FRONT PANEL OF MODEL 263A

- Galvanostatic Scan, with a gradual ramp between the three levels of cell current. You can program the rate of change ("SCANRATE") of the current between levels.

- Galvanostatic Step, with sharp steps between the three levels of cell current.

The lengths of time for which each of the three levels of potential or current are held constant during the experiment are called T Init, T Mid, and T Final. Each time can be separately programmed (or omitted entirely).

Note that the Model 263A does not apply a linear scan, but rather a "staircase" scan. However, as long as the individual steps are very small relative to the range scanned, linear scan theory will apply to a close approximation. The nominal step size is the scan rate/250, or 250 μV, whichever is larger. Under remote control via the rear panel GPIB or RS-232 port, steps as small as 25 μV (MR = 1), or 2.5 μV (MR = 0), can be attained as explained in the *Model 263A Command Set Handbook*).

Example: With a scan rate of 1 V/s, the step size will be 4 mV, because 1000/250 = 4.

5.3 CONTROLS AND INDICATORS

In local control (when you are not controlling the Model 263A from a separate computer), you communicate with the Model 263A using the controls and liquid crystal display (LCD) on its front panel (Figure 5-1). The controls – a large knob and 13 push-button keys – are used to set the test parameters and select the operations to be performed. The LCD panel provides continuous, real-time information on the experiment, including the parameters initially set and the cell potentials and currents measured or applied as the experiment progresses. This section describes the functions of the controls, LCD, and indicator lights.

POWER Switch: The POWER switch, when pushed in, provides ac power to the instrument. In the off position, it interrupts both sides of the ac supply to the instrument.

OVERLOAD Indicators: The *I OVERLOAD* indicator lights if the working electrode current exceeds two times the full-scale current range. This does not mean that the cell control loop is out of control. For example, suppose the Model 263A were programmed to establish a potential of +0.752V, and that under the established conditions, a cell current of 0.5 mA occurred. If the current range were 1 μA, 10 μA, or 100 μA, the current monitoring circuits would be driven to the limit and the I OVERLOAD indicator would light, even though the cell would still be controlled at +0.752V.

Unlike with the I OVERLOAD indicator, a lighted *E OVERLOAD* indicator always means that the cell control loop is out of control. That is, the control amplifier has reached its maximum compliance voltage of $\pm$20 V, and the potential of the working electrode with respect to the reference electrode is not as programmed. This could result from a connection error (open cell connection), an electrode problem, or an unacceptably high solution resistance.

CELL Indicator: This indicator lights whenever the internal cell relay is closed. The cell relay is controlled either by the CELL switch or by the CELL command sent from an attached computer.

CELL Switch: The CELL pushbutton controls the cell relay, which determines whether the control amplifier is connected to the cell. When the CELL switch is off (CELL indicator not illuminated), the control amplifier is disconnected from the cell and there is zero cell current, allowing the cell connections to be made safely. When the CELL switch is on (CELL indicator lit), the control amplifier is connected to the cell.

The cell relay also can be controlled by a software command from an attached computer.

The cell control loop remains controlled even when the cell isn't on (regardless of whether CELL TYPE is set to REAL or DUMMY). Having the cell off does not cause an overload condition.

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MODE Key: This pushbutton selects the basic operating mode of the Model 263A – POTENTIOSTAT or GALVANOSTAT – and selects either a discrete step or a gradual scan between applied voltage or current levels. An indicator lamp lights to show whether the POTENTIOSTAT or GALVANOSTAT mode has been selected, and the appropriate menu screen appears on the LCD panel. Figure 5-2 shows the sequence in which the experiment menu screens appear when you repeatedly press the MODE key.

Note that you cannot switch between potentiostatic and galvanostatic operation during an experiment, as this would require basic changes in the entire system configuration. Thus, when the cell is on, you cannot change between potentiostatic and galvanostatic menus. If the [Galvanostatic Step] menu is displayed, pressing the MODE key will switch to the [Galvanostat] menu. Similarly, if the [Potentiostatic Step] menu is displayed, pressing the MODE key will switch to the [Potentiostat] menu.

The MODE key also can be used to move from the [System Default] menu screen on the LCD display to one of the potentiostat or galvanostat menus (see Figure 5-3). The MODE key is disabled during remote-control operation.

SYSTEM Key: In local control, the SYSTEM pushbutton allows you to access the [System Interface] menu screen on the LCD panel from any potentiostat or galvanostat menu (see Figure 5-3). It also moves you from the [System Interface] menu to the [System Default] menu and from the [System Default] menu to a potentiostat or galvanostat menu.

LCD Display: The four-line liquid crystal display provides continuous information on the experiment in progress, including the parameters set and the voltage and current measurements being taken. The information shown depends on which of the several available menu screens is displayed. In local control, the MODE and SYSTEM keys above the display allow you to cycle through the screens. Section 5.4 describes the features of the LCD panel and the menu screens it can display.

In the event of a system malfunction, the LCD panel will display an error message describing the problem and sometimes directing the operator to take the required action. For an explanation of these error messages, refer to Section 5.10. Remote-control software error messages are explained in the *Model 263A Command Set Handbook*.

F1 Through F5 Function Keys: In local control (when you are controlling the Model 263A from its front panel), these "soft" keys perform various actions before and during an experiment. You use them to start your experiment and control its progress. You can also use them to recalibrate the instrument (see Section 5.8) and reset the experiment parameters to their default values (Section 5.7).

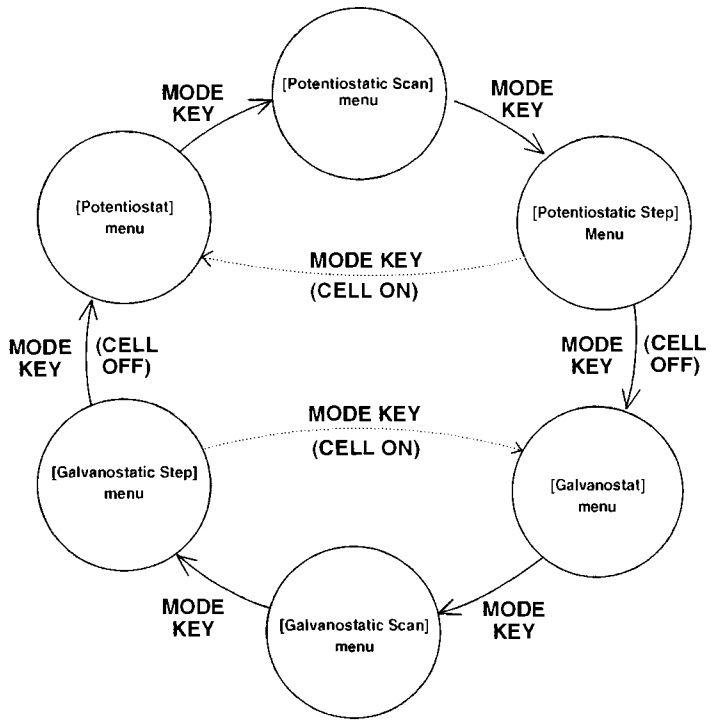


Figure 5-2. ORDER OF MENU SCREENS THAT APPEAR WHEN YOU PRESS THE MODE KEY FROM ANY POTENTIOSTATIC OR GALVANOSTATIC MENU SCREEN

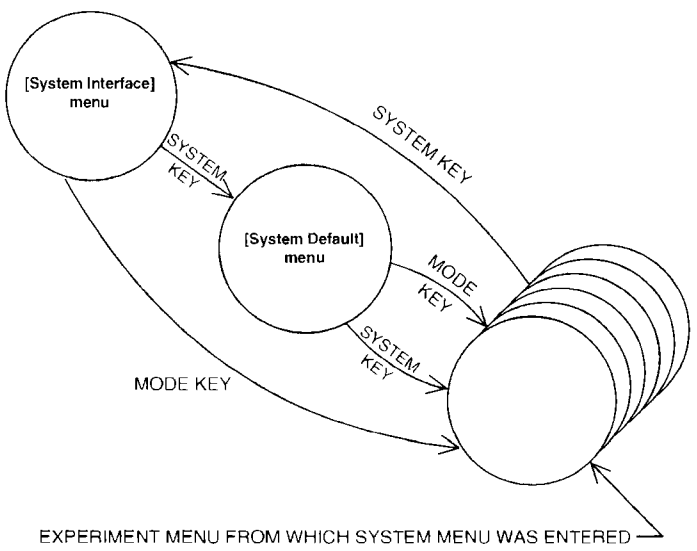


Figure 5-3. MOVING AMONG THE SYSTEM, FUNCTION, AND POTENTIOSTATIC OR GALVANOSTATIC MENU SCREENS

The function assigned to each key is displayed on the bottom line of the LCD panel. These functions may change if you switch to another menu screen.

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When the Model 263A is under control of an external computer, F1 through F4 have no functions assigned. Pressing F5 will return you to local control and bring up either the [Potentiostat] or [Galvanostat] menu screen.

VALUEKnob: In local control, this knob sets the value of any experiment parameter selected with the PREV and NEXT PARAMETER keys. The parameter being set is displayed on the third line of the LCD display.

The VALUE knob provides tactile feedback and has a speed sensing feature to make parameter setting easy and intuitive. As you would expect, the faster you turn the knob the faster the parameter value changes. But note that the relationship between knob speed and value change does not remain linear. Above a certain knob speed the processor "shifts gears" and changes to a higher ratio of parameter change to knob speed. This allows you to make large changes in the value of a parameter by spinning the knob rapidly.

To prevent someone from inadvertently changing the value of a parameter by brushing against the VALUE knob, you can disable the knob after using it. You can specify the period of time it waits after its last use before becoming inactive (the "timeout" period). The Knob Timeout parameter is set from the [System Interface] menu screen. See Section 5.5 for a more detailed explanation of the timeout parameter and how to set it.

PREV and NEXT PARAMETERKeys: In local control, these keys select the next available or the last available experiment parameter for display. The parameter selected is displayed on the third line of the LCD panel. The value of the displayed parameter can then be set with the VALUE knob.

Full-Scale Current Range Indicators, 100 mA (1 A) through 100 nA: These indicator lamps monitor the Model 263A's full-scale current range. One of the seven available current ranges is always selected. A cell current equal to the selected current range gives 1 V at the I OUTPUT connector. Note that the 100 mA (1A) indicator is yellow if the current range is 100 mA and red if it is 1 A. *The 1 A current range is only available of the 94 Option has been installed.*

In local control, the full-scale current range can be selected either manually by the operator or automatically by the Model 263A. In the Manual mode, the range is selected with the arrow keys under the indicator lamps. The ↑ ↑ key moves the selection upwards through the available ranges. The ↓ ↓ key moves it downwards. The Auto mode is described below under **AUTO Switch and Indicator**.

↑ ↑ Pushbutton: In the Manual current ranging mode (under local control), this key is used to increase the full-scale current range of the Model 263A. Pressing this key steps the full-scale current range to the next higher range available. The current range set is shown by illumination of the corresponding indicator lamp. This key has no effect when AUTO current ranging is on.

↓ ↓ Pushbutton: In the Manual current ranging mode (under local control), this key is used to decrease the full-scale current range of the Model 263A. Pressing this key steps the full-scale current range to the next lower range available. The current range set is shown by illumination of the corresponding indicator lamp. This key has no effect when AUTO current ranging is on.

AUTO Switch and Indicator: In the Potentiostat mode (local control), the AUTO switch is used to select manual or automatic adjustment of the full-scale current range. Pushing the switch toggles between the auto and manual modes. The AUTO Indicator lamp is lit when AUTO is selected.

Note that automatic current ranging is restricted to operation in the Potentiostat mode. It cannot be selected in the Galvanostat mode.

In the AUTO current range mode, the Model 263A automatically seeks the full-scale current range that causes the I OUTPUT potential to be between 15% and 190% of full scale; that is, between 150 mV and 1.9 V. If cell current is less than 150 nA, the 100 nA range will be selected. If the instrument is on the 10 mA range and cell current is greater than 19 mA, the 100 mA range will be selected. This process occurs after each point is acquired, with the constraint that it can only change one range at a time. Data could be lost if the data amplitude is changing faster than it can be tracked by the auto-ranging function.

You can set a limit to the automatic current ranging by adjusting the I AUTO LIMIT parameter on the [System Default] menu (see Section 5.6). The full-scale current range will not go below the I AUTO LIMIT you set.

The I/E Filter (if selected) can cause unpredictable effects on your experiment if AUTO current ranging is in use. If I/E FILTER = ON is selected (from the [System Default] menu screen), we recommend manual current ranging. See the discussion of the I/E FILTER parameter in Section 5.6.

5.4 USING THE LCD MENU SCREENS

In local control, the LCD panel and its associated controls provide the means for communication between the researcher and the Model 263A. The researcher first sets the experiment parameters he or she needs on the appropriate menu screens, using the VALUE knob. He then presses F3 to start the experiment. At any time during the scan he can reverse its direction, suspend its progress and then restart it, or stop it entirely.

The LCD panel can display four lines of alphanumeric characters. The information shown on these lines depends on which menu screen is displayed. Under local control,

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eight screens are available: the [System Interface] and [System Default] screens, three potentiostat screens, and three galvanostat screens. The MODE and SYSTEM keys above the display allow you to cycle through the screens.

When the Model 263A is under control of an external computer, the LCD panel changes to the REMOTE screen. All the front-panel controls except the CELL and POWER switches and the F5 function key are disabled, but the indicator lamps continue to function. Pressing F5 (now called the LOCAL key) will return you to local control and bring up either the [Potentiostat] or [Galvanostat] menu screen. You can use the MODE key from either of these screens to move to any other local-control menu screen.

Note: We recommend that you do not mix front-panel and remote operations. Please see the note in Section 5.1.

Common Screen Elements
The top line of the LCD panel (Figure 5-4) shows the name of the current menu screen in brackets ("REMOTE" is displayed when the instrument is under control of an external computer). On the [System Interface] menu, the top line also shows the name of the instrument and the version of the firmware installed.

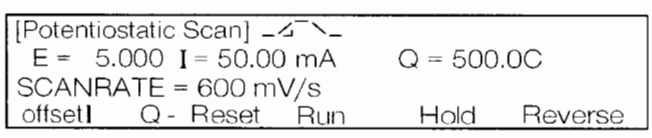


Figure 5-4. TYPICAL LCD MENU SCREEN

The second line of the display continuously shows, on the left side of the line, the measured and corrected cell potential as the experiment progresses. If a cell potential overload condition occurs (see OVERLOAD Indicators in Section 5.3), "E = OVERLOAD" will be displayed.

The center of this line continuously shows the actual cell current (I) without I Offset applied. (If a current overload condition occurs, "I = OVERLOAD" will be displayed here. This means that the current is too large to be measured. It does not mean that the cell control loop is out of control.)

The right side of this line displays either the current with offset applied (i) or the total charge (Q) that has passed through the cell since the beginning of the experiment. To display the value of i or Q, press F1 (see "Bottom Row Functions" below).

Setting Parameters: In local control, the third line of the display shows one of several experiment or communications parameters that can be set by the operator. To change the value of the displayed parameter, simply turn the VALUE knob. The PREV and NEXT PARAMETER keys, located under the VALUE knob, allow you to display in turn each of the adjustable parameters. The value of any parameter displayed on the third line can be reset with the VALUE knob.

A useful feature of the Model 263A is that you can change many of the parameters at any time during a scan, as well as before the scan begins. You can change the following parameters while the scan is in progress:

E INIT	E MID	E FINAL
I INIT	I MID	I FINAL
T INIT	T MID	T FINAL
SCANRATE	CYCLES	CELL@END

When the Model 263A is under control of an external computer, the third line of the REMOTE screen may be blank, it may display up to 40 characters of text typed on the computer following the TYPE command, or it may display a communications error message (see Section 5.10).

Bottom Row Functions: The fourth (bottom) line of the display shows the function currently assigned to each of the "soft" function keys (F1 through F5). You will note that the functions of some keys vary according to the menu screen displayed. In some menus, not all of the function keys are used.

5.5 SETTING COMMUNICATIONS PARAMETERS

The [System Interface] menu screen is used to set up certain basic operating parameters for your system. All but one of these parameters (KNOB TIMEOUT) concern communications between the instrument and a host computer, and do not have to be set if you are operating from the front panel of the Model 263A. These parameters can be displayed sequentially on the menu (line 3) by pressing the PREV and NEXT PARAMETER keys. After you select a parameter, you may change its setting with the VALUE knob.

The SYSTEM key allows you to access the [System Interface] menu screen from any potentiostatic or galvanostatic menu. It also moves you from the [System Interface] menu to the [System Default] menu and from the [System Default] menu to a potentiostatic or galvanostatic menu (see Figure 5-3).

The MODE key is used to move directly from the [System Interface] menu screen to one of the potentiostatic or galvanostatic menus, as shown in Figure 5-3. (It is also used to move between the potentiostatic and galvanostatic experiment modes).

The operating parameters that can be displayed on line 3 of the [System Interface] menu (and adjusted with the VALUE knob) are listed below. Details of these parameters are given in Chapter 8 of this manual and in the appendixes of the *Model 263A Command Set Handbook*.

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Note that when you are controlling the Model 263A from a host computer via the RS-232 bus, the Baud rate, number of data bits, parity, and number of stop bits must be the same for both the Model 263A and the serial port on the host computer.

- GPIB ADDR (1 to 30). The normal GPIB address setting for use with EG&G Instruments software is 14. If you are using other software, see the software manual for its requirements.

- TERMINATOR (CR or CRLF). The terminator required for GPIB communications with IBM PC-compatible computers running EG&G Instruments software is CR.

- GPIB ECHO (OFF or ON). During normal operation, GPIB ECHO should be set to OFF. When it is ON, every character transmitted or received via the GPIB port will be echoed to the RS-232 Interface Connector. This feature is particularly useful when developing programs. If a "dumb" CRT terminal is connected to the RS-232 Interface, the programmer will see all communications on the CRT.

- SERIAL BAUD (110/300/600/1200/2400/4800/9600/19200). This sets the Baud rate for RS-232 communications. Hardware handshaking is recommended for the higher settings.

- SERIAL PRTY (NONE, EVEN, ODD). This determines parity checking for RS-232 communications. Parity maintenance provides a direct way of detecting garbled data if the computer is programmed to only accept words having the selected parity. If a word having incorrect parity is read, it is a message to the computer that data loss is occurring.

- SERIAL BITS (7 or 8). This sets the number of bits in the data word in RS-232 communications. Eight bits are required for binary data loads and dumps (the eighth bit is the sign bit).

- SERIAL STOP (1 or 2). This sets the number of stop bits in RS-232 communications. Two stop bits give more reliable communications at high baud rates, but slow the data transfer.

- SERIAL ECHO (OFF or ON). Normally OFF. If ON is selected, each character received via the RS-232 port will be echoed back to the character source. The RS-232 echo is normally used only when the Model 263A is connected to a CRT terminal.

  Note: Do not confuse this function with the GPIB ECHO.

- KNOB TIMEOUT (DISABLED, 1 to 120 s). To prevent someone from inadvertently changing an experiment parameter by brushing against the VALUE knob, you can disable the knob after using it. You can specify the period of time it waits after its last use before becoming inactive (the "timeout" period). At the end of the timeout period the knob will have no effect on parameter settings, and the LCD panel will display "KNOB TIMEOUT HIT ANY KEY." Press any key to reactivate the knob. When DISABLED is selected, the knob remains active and will not time out.

5.6 COMMON EXPERIMENT PARAMETERS

The [System Default] menu screen allows you to view and, if necessary, adjust parameters that normally do not require resetting between experiments. As in the other menus, these parameters can be displayed sequentially on the menu (line 3) by pressing the PREV and NEXT PARAMETER keys. After you select a parameter, you may change its setting with the VALUE knob.

The SYSTEM key allows you to access the [System Default] menu screen from the [System Interface] menu. It also moves you from a system menu to a potentiostatic or galvanostatic menu (see Figure 5-3). The MODE key also can be used to move from the [System Default] menu screen to one of the potentiostatic or galvanostatic menus.

The experiment parameters that can be displayed on line 3 of the [System Default] menu (and adjusted with the VALUE knob) are listed below. Details of these parameters are given in Chapter 8.

- CELL TYPE (REAL or DUMMY). Use REAL for normal operation with a cell. In the DUMMY position, an internal 10 kΩ, .01% resistor is substituted for a connected cell. The dummy load is useful for determining that the Model 263A is functioning correctly.

- EXT INPUT (OFF or ON) determines whether the front-panel EXT INPUT connector will accept an applied signal without applying a scaling factor to it. Use ON if the applied signal has a suitable amplitude and you do not wish to scale it. OFF is used either when no signal is to be applied, or when an applied signal is too large or too small and you wish to scale it with the OSC GAIN parameter.

  Note that both EXT INPUT and OSC IN (see below) cannot be set to ON at the same time. If you are unsuccessful in turning one on, check that the other is off.

- OSC IN (OFF or ON) determines whether the front-panel EXT INPUT connector will accept and scale an applied signal. Use ON if the ap-

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plied signal is too large or too small and you wish to scale it by a factor of 0.02.0.2, or 2.0. OFF is used either when no signal is to be applied, or when an applied signal is of a useful amplitude with unity gain (in that case, use EXT INPUT = ON).

Note that both EXT INPUT and OSC IN cannot be set to ON at the same time. If you are unsuccessful in turning one on, check that the other is off.

If you set OSC IN = ON, press the NEXT PARAMETER key once and set OSC GAIN (see below).

- OSC GAIN (X.02, X.2, or X2) sets the OSC GAIN factor to one of the three available values: 0.02, 0.2, or 2.

- LINE SYNC (OFF or ON) determines whether data acquisition will be synchronized with the power-line frequency. When ON is selected, the sample is taken when the next line-frequency zero crossing occurs. This occurs every 16.6 ms (60 Hz power) or every 20 ms (50 Hz power). If LINE SYNC = OFF, samples are taken at the rate determined by the internal time base of 4 ms. The default state is OFF.

- I/E FILTER (OFF or ON). ON selects two low-pass filters which filter the signal input to the A/D converter. One of these filters has a sharp cutoff at 5.3 Hz. (In remote operation, a 590 Hz low-pass filter is also available under software control.)

  The second low-pass filter selected by setting I/E FILTER = ON consists of a roll-off capacitor that reduces the bandwidth of the I/E converter. The effect of this capacitor on bandwidth depends on the current range selected. Cut-off frequencies for this filter at each current range are given in Chapter 8, Section 8.2, under "Filters."

  Unlike the 5.3 Hz filter, the roll-off capacitor filters the signal at the I OUTPUT connector. This capacitor is often helpful when making low-current measurements on large electrodes because it reduces the noise at I OUTPUT. The disadvantage of using the filter is that the I OUTPUT response time is degraded.

  It may be advantageous to synchronize data acquisition with the power line frequency when filtering. This can be done by setting LINE SYNC = ON (see LINE SYNC above).

  The I/E filters can cause unpredictable effects on your experiment if AUTO current ranging is in use. If I/E FILTER = ON is selected, we recommend manual current ranging.

- BANDWIDTH (HI STABILITY or HI SPEED) sets the bandwidth/stability parameter. For the fastest response, choose HI SPEED. For extreme stability, choose HI STABILITY.

- I AUTO LIMIT sets the most sensitive current range to which the Model 263A can automatically range.

- UPDATE Q W/CELL OFF (NO or YES). The final current integral (Q), in coulombs, can be displayed, with continuous updating as the measurement progresses. The final current integral is the net charge (including I Offset) that has passed through the cell since the beginning of the experiment while the cell was on.

  In a perfect instrument and cell system, Q of course could not change when the cell is disconnected, because no cell current would flow. But in a real system, no matter how precise, some small charge may appear as a result of drift and other circuit variations. Thus, setting UPDATE Q W/CELL OFF = NO prevents charge from accumulating when the cell is off. Choosing YES allows it to accumulate, and might help to estimate drift errors.

5.7 RESETTING PARAMETERS TO DEFAULT VALUES

In certain circumstances it may be desirable to return to the default values of the experiment parameters, and possibly the communications parameters also. For example, the default parameters are stored in battery-backed RAM. If the battery has been replaced, the parameters will have to be reset, and it is easiest to start from the default values.

There are two ways you can return to the default values of the parameters. One method resets only the experiment parameters—those that appear on the [System Default] menu and the potentiostatic and galvanostatic menus. It does not alter the parameters on the [System Interface] menu (communications parameters and KNOB TIMEOUT). The second method resets *all* parameters, including those on the [System Interface] menu. Both procedures are described in the following.

To reset only the experiment parameters: With the instrument running, press the SYSTEM key to display the [System Interface] menu screen. Then, press "Reset" (F2). The following query will be displayed on the LCD panel:

RESET SYSTEM? . . .

If you press YES (F4), all experiment parameter selections will be cleared and default values will be

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restored. If you press NO (F5), the parameters will not be reset and the SYSTEM menu screen will reappear.

To reset all parameters: With the instrument off, hold down any front-panel key and press the POWER switch. The following message will be displayed on the LCD panel:

KEY HELD DURING POWER UP
SYSTEM RESET PERFORMED
(third line is blank)
(fourth line is blank)

When the message clears, all default parameter values will be restored and the Model 263A will be ready to run your experiment.

Note that the experiment parameters are not reset when the power is cycled. When the Model 263A powers down, all parameter values and selections are retained. When it is powered up, most of the values in effect at the time of power shutdown are restored.

Note also that the foregoing two reset procedures do not clear the calibration values or recalibrate the instrument. If the battery has been replaced, you will also have to recalibrate. Refer to Section 5.8 below for the calibration procedure.

5.8 CALIBRATING THE MODEL 263A

In the [System Interface] menu, F1 is assigned the Calib (Calibrate) function. This function is used to recalibrate the Model 263A. Calibration values are stored in battery-backed RAM. If the battery has been replaced, use this procedure to recalibrate the instrument. Before recalibrating, allow the instrument to warm up for at least ½ hour.

To recalibrate the instrument, press the SYSTEM key to display the [System Interface] menu screen, and then press CAL (F1). The following query will be displayed on line 3 of the LCD panel:

CALIBRATE SYSTEM? . . .

If you press NO (F5), recalibration will not proceed and the [System Interface] menu screen will reappear. If you press YES (F4), the CALIBRATING screen is displayed. As each step in the calibration procedure is performed in sequence, its number and name appear on the second line of the screen. The third line will contain further information during some calibration steps; during others it will be blank. The fourth line shows the constantly changing calibration parameter values.

If calibration fails, for any reason, the second line display (step number and name) will freeze, the third line will now say FAIL, and the fourth line will say HIT ANY KEY TO CONTINUE. Pressing any key will leave the CALIBRATING screen and return to the [System Interface] menu. You may now run experiments, but the results will be suspect because you won't know how far the instrument is out of calibration.

Therefore, if calibration fails, we recommend that you not take the advice on the fourth line and "hit any key." Instead, leave it in the CALIBRATING screen and call the EG&G Instruments Service Department (609-530-1000) to report the number and name of the step that failed. This information will continue to be shown on the second line of the display. The service technician will diagnose the problem and advise you as to what action to take.

5.9 DETERMINING INSTALLED OPTIONS

In the [System Interface] menu, F3 is assigned the Options function. The Model 263A is designed for easy expansion to accommodate future enhancements now under development or envisioned. As these optional features become available, the Options function will allow a user or service technician to determine easily exactly how the system is configured.

To see which options are installed, press the SYSTEM key to display the [System Interface] menu screen, and then press "Options" (F3). A string of numbers separated by spaces will be displayed on the third line of the LCD panel. Each number is in the range 90 to 99 and indicates the presence of an installed option. The number 90 indicates the presence of the basic system PC boards and should always be displayed. Instruments without options installed will display the number 90 only. If Option 99 is installed, the Float switch status will be displayed in parentheses.

On line 4 of the LCD panel, the prompt

HIT ANY KEY TO CONTINUE

will be displayed. After you have read the display of options, press any key to return to the [System Interface] menu.

5.10 ERROR MESSAGES

Certain conditions that interfere with normal operation will cause error messages to be displayed on the LCD panel. The message will give a warning statement about the type of error, information about the cause (sometimes in a numeric code), and usually advice about what action to take.

Note that errors indicated on the LCD panel are not related to the software error codes that you can display on the monitor of a host computer with the ERR command.

The following shows the error message screens that may appear on the LCD panel when a problem occurs.

CATASTROPHIC FAILURE
MISSING BOARDS *(means it can't find a PC board)*

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(error code numbers will appear here)
SYSTEM HALTED

CATASTROPHIC FAILURE
BAD CRC ON BOARDS *(means a memory failure)*
(error code numbers will appear here)
SYSTEM HALTED

If either of the foregoing two error message screens is displayed, call the EG&G Instruments Service Department (609-530-1000) and report the error code displayed on line three. The service technician will diagnose the problem and advise you as to what action to take.

BAD BOARD ID SHADOW CRC
BATTERY PROBABLY DEAD
CALIBRATION REQUIRED
HIT ANY KEY TO CONTINUE

The Model 263A is equipped with a long-life lithium battery that enables retention of the calibration settings in memory. The battery has a life of approximately two to five years, depending on how much you use the instrument. If the foregoing screen is displayed, you will probably have to send the instrument to an authorized EG&G Instruments repair facility for battery replacement. You cannot replace the battery yourself.

You can use the instrument without the battery, but you will have to perform the calibration procedure every time you power up. If you need to complete some work first before you send the unit, you can calibrate it and continue with your work. Before calibrating, allow the instrument to warm up for at least ½ hour.

To recalibrate, first press any key to clear the error message. Then, press the SYSTEM key to bring up the [System Interface] menu screen. Follow the calibration instructions in Section 5.8.

If the error message reappears, call the EG&G Instruments Service Department (609-530-1000) and report it. The service technician will diagnose the problem and advise you as to what action to take.

BOARD(s) CHANGED: *(new bd has different rev. level)*
SYSTEM: *(number)* OPTIONS: *(number)*
MAY REQUIRE CALIBRATION
HIT ANY KEY TO CONTINUE

If the foregoing error message screen is displayed, you should recalibrate the instrument. Before recalibrating, allow the instrument to warm up for at least ½ hour.

To recalibrate, first press any key to clear the error message. Then, press the SYSTEM key to bring up the [System Interface] menu screen. Follow the calibration instructions in Section 5.8.

REMOTE
E= *X.XXXV* I= *X.XXX*mA Q=*X.XXX* c
(blank or TYPE message)
COMM ERROR *XX* LOC

(means PC communications have failed)

In the foregoing error message screen, the words "COMM ERROR" on the fourth line are followed by a two-digit hexadecimal error code.

If this screen is displayed either (a) when you turn on the Model 263A or (b) while you are controlling the Model 263A from a host computer, call the EG&G Instruments Service Department (609-530-1000) and report the error code. The service technician will diagnose the problem and advise you as to what action to take. Then, press LOC (F5) to clear the error message.

If the foregoing COMM ERROR message screen is displayed while you are trying to establish communications between a host computer and the Model 263A *via the RS-232 bus*, first press LOC (F5) to clear the error message. Then, check that the following communications parameters are set correctly on the Model 263A to match the settings on the computer:

- Baud rate
- Number of data bits
- Number of stop bits
- Parity
- Terminator (usually should be CR)

After you have reset any communications parameters that don't match the computer software requirements, send an Action command from the computer to the Model 263A. If the command is executed, the problem has cleared. If the error message returns, call the EG&G Instruments Service Department (609-530-1000) and report the error code.

The following is not an error message but a cautionary message to warn you that experiment and communications parameters have been reset to their default values:

KEY HELD DURING POWER UP
SYSTEM RESET PERFORMED
(third line is blank)
(fourth line is blank)

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Chapter 6
Running Potentiostatic Experiments

6.1 INTRODUCTION

This chapter shows you how to set the experiment parameters that are unique to potentiostatic operation, and how to run your potentiostatic experiments.

But before you proceed any further, it is important to become familiar with the information in some of the previous chapters. Chapter 2 describes important safety precautions for operating this instrument. Chapter 3 shows you how to connect the instrument to your electrochemical cell and any other equipment you may be using with it. Chapter 4 takes you through a sample session with the instrument to introduce you to its operation.

Chapter 5 is particularly important, as it describes the functions of the controls and indicator lights on the front panel. It also shows you how to move among the eight menu screens that are available on the LCD display. It then explains how to set up the basic experiment parameters that seldom require changing. (These are set on the [System Default] and [System Interface] menus.)

Chapter 5 also shows how to set the communications parameters necessary for controlling the instrument from a host computer. Instructions for operating the unit remotely via either the RS-232 or GPIB (IEEE-488) interface port are provided in the separately bound *Model 263A Command Set Handbook*.

6.2 POTENTIOSTATIC MODES

Under front-panel control, the Model 263A can hold the working electrode potential fixed at a single level during the experiment, or it can vary the potential between three separate programmable levels, called E Init, E Mid, and E Final.

The transition between the three levels can be in the form of either a gradual ramp or "scan" between levels, or a sudden sharp "step."So there are actually three modes of potentiostatic operation available:

- Potentiostatic with a fixed potential [Potentiostat], where the potential at the working electrode is held constant.

- Potentiostatic Scan [Potentiostatic Scan], with a gradual ramp between the three levels of working electrode potential. You can program the rate of change ("SCANRATE") of the potential between levels.

- Potentiostatic Step [Potentiostatic Step], with sharp steps between the three levels of working electrode potential.

The lengths of time for which each of the three levels of potential are held constant during the experiment are called T Init, T Mid, and T Final. Each time can be separately programmed (or omitted entirely).

To run a potentiostatic experiment from the front panel, first set up the basic parameters as described in Chapter 5. Then, press the MODE key as many times as necessary to display the menu screen for the potentiostatic mode you wish to use: [Potentiostat], [Potentiostatic Scan], or [Potenitostatic Step].

After you select the appropriate menu screen, use the PREV and NEXT PARAMETER keys to display each experiment parameter in turn, as described in Chapter 5. The parameters used in each potentiostatic mode are described in the following sections. More detailed explanations are given in Chapter 8. If the default value of any parameter is not satisfactory for your experiment, use the VALUE knob to change it.

6.3 SETTING FIXED-POTENTIAL EXPERIMENT PARAMETERS

The [Potentiostat] menu allows you to set the following experiment parameters used in steady-state potentiostatic operation, where the potential at the working electrode is held constant with respect to the reference electrode.

- POTENTIAL at the working electrode.

- IR MODE (OFF or ON and, if ON, whether Positive Feedback (PFIR) or Current Interrupt (IRUPT) iR compensation is to be used).

- PFIR COMP. When Positive Feedback iR compensation is selected, you specify the amount of resistance here (in ohms).

- IRUPT T1 sets the increment of time between the interruption of current to the cell at the start of a periodic Current Interrupt iR Compensation cycle and the first voltage sampling. This voltage sampling, in conjunction with the second sampling at IRUPT T2, is used to extrapolate back to the interrupt potential in determining the Current Interrupt iR Compensation correction factor. The range is 10 μs to 2000 - (IRUPT T2) μs. The default value is 10 μs on the 100 mA range (also on 1 A range if 94 option is installed) and 75 μs on all other ranges. Note that IRUPT T1 + IRUPT T2 may not exceed 2000 μs. Note also that IRUPT T1 *can be set independently for each current range*.

- IRUPT T2 sets the increment of time between the first voltage sampling during a periodic

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Current Interrupt iR Compensation cycle and the second voltage sampling. The voltage samplings are used to extrapolate back to the interrupt potential in determining the Current Interrupt iR Compensation correction factor. The range is 10 μs to 2000 - (IRUPT T1) μs. The default value is 10 μs on the 100 mA range (and on the 1 A range if 94 option is installed) and 75 μs on all other ranges. Note that IRUPT T1 + IRUPT T2 may not exceed 2000 μs. Note also that IRUPT T2 *can be set independently for each current range*.

- IRUPT (Intermittent Current Interrupt) sets the number of points between current interrupts in Current Interrupt iR Compensation. The range is 1 to 32,767 points, with a default of 5 points. As the time base used in local operation is 4 ms, 5 points is equal to 20 ms.

- IRPC (Current Interrupt Percent Correction) is the percent of correction applied in Current Interrupt iR Compensation. The range is 0% to 200%. The default value is 100%.

- I OFFSET (current offset) allows you to specify a current value to be mathematically subtracted from the cell current before it is displayed. The result of this subtraction is displayed as "i" and accumulated over time as "Q". That is, i = I - (I OFFSET), where I OFFSET can have a positive or negative value.

  Note that there is no actual current subtraction, only a numerical calculation. Thus current offset does not affect the potential at the I OUTPUT connector.

  The offset is specified in units of current. The offset range is $\pm$2 times the full-scale current range in effect at the time I OFFSET is set.

These parameters are explained in greater detail in Chapter 8. After you have set them as required for your experiment, follow the directions in Section 6.6 for running your experiment.

6.4 SETTING SCANNED-POTENTIAL EXPERIMENT PARAMETERS

The [Potentiostatic Scan] menu is used to set the following experiment parameters for the Potentiostat Scan mode. In this mode, the Model 263A varies the potential of the working electrode in a gradual sweep or ramp between steady-state levels.

- E INIT, the potential at the working electrode during the first steady-state stage of a scan or step sequence.

- E MID, the potential at the working electrode during the second steady-state stage (sometimes called the "vertex") of a scan or step sequence.

- E FINAL, the potential at the working electrode during the third steady-state stage of a scan or step sequence.

- T INIT, the length of time that E INIT is maintained at the working electrode.

- T MID, the length of time that E MID is maintained at the working electrode.

- T FINAL, the length of time that E FINAL is maintained at the working electrode.

- SCANRATE, the rate of change of the potential at the working electrode between E INIT and E MID, and between E MID and E FINAL.

- CYCLES allows selection of three different scan or step sequences: HALF CYCLE, FULL CYCLE, and CONTINUOUS. The cycle setting determines the number of separate steady-state potential levels that will be used at the working electrode during your experiment.

- CELL@END determines the working electrode potential at the end of the scan or step sequence. If you select OFF, the cell will be turned off (counter electrode connection interrupted) at the end of the programmed cycle. If you select E INIT, the working electrode potential will be returned (stepped sharply) to E INIT.

  If you select E FINAL, the working electrode potential remains at its last value in the sequence. In the FULL CYCLE mode, this is E FINAL. But note that in HALF CYCLE operation, the last potential level reached in the sequence actually is E MID.

  Also note that in the CONTINUOUS cycle mode, the experiment doesn't end until the STOP key (F3) is pressed. At that point, the working electrode potential returns to E INIT.

- IR MODE (OFF or ON and, if ON, whether Positive Feedback (PFIR) or Current Interrupt (IRUPT) iR compensation is to be used).

- PFIR COMP. When Positive Feedback iR compensation is selected, you specify the amount of resistance here (in ohms).

- IRUPT T1 sets the increment of time between the interruption of current to the cell at the start of a periodic Current Interrupt iR Compensation cycle and the first voltage sampling. This voltage sampling, in conjunction with the second sampling at IRUPT T2, is used to extrapolate back to

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the interrupt potential in determining the Current Interrupt iR Compensation correction factor. The range is 10 μs to 2000 - (IRUPT T2) μs. The default value is 10 μs on the 100 mA range (and on 1 A range if 94 option is installed) and 75 μs on all other ranges. Note that IRUPT T1 + IRUPT T2 may not exceed 2000 μs. Note also that IRUPT T1 *can be set independently for each current range.*

- IRUPT T2 sets the increment of time between the first voltage sampling during a periodic Current Interrupt iR Compensation cycle and the second voltage sampling. The voltage samplings are used to extrapolate back to the interrupt potential in determining the Current Interrupt iR Compensation correction factor. The range is 10 μs to 2000 - (IRUPT T1) μs. The default value is 10 μs on the 100 mA range (and on 1 A range if 94 option is installed) and 75 μs on all other ranges. Note that IRUPT T1 + IRUPT T2 may not exceed 2000 μs. Note also that IRUPT T2 *can be set independently for each current range.*

- IRUPT (Intermittent Current Interrupt) sets the number of points between current interrupts in Current Interrupt iR Compensation. The default is 5 points. As the time base used in local operation is 4 ms, 5 points is equal to 20 ms.

- IRPC (Current Interrupt Percent Correction) is the percent of correction applied in Current Interrupt iR Compensation. The range is 0% to 200%. The default value is 100%.

- I OFFSET (current offset) allows you to specify a current value to be mathematically subtracted from the cell current before it is displayed. The result of this subtraction is displayed as "i" and accumulated over time as "Q". That is, i = I - (I OFFSET), where I OFFSET can have a positive or negative value.

  Note that there is no actual current subtraction, only a numerical calculation. Thus current offset does not affect the potential at the I OUTPUT connector.

  The offset is specified in units of current. The offset range is $\pm$2 times the selected full-scale current range.

These parameters are explained in greater detail in Chapter 8. After you have set them as required for your experiment, follow the directions in Section 6.6 for running your experiment.

6.5 SETTING STEPPED-POTENTIAL EXPERIMENT PARAMETERS

In the Potentiostat Step mode, the Model 263A varies the potential of the working electrode in sharp steps between steady-state levels. The experiment parameters that can be set from the [Potentiostatic Step] menu are the same as those set from the [Potentiostatic Scan] menu (see Section 6.4 above), except that there is no SCANRATE setting. After you have set the parameters as required for your experiment, follow the directions in Section 6.6 for running your experiment.

6.6 RUNNING YOUR EXPERIMENT

After you have connected your equipment (as described in Chapter 3) and set all required parameters (as described in Chapter 5 and this chapter), you are ready to begin your experiment. You will start the experiment and control its progress with the "soft" function keys, F1 through F5, below the LCD panel.

The functions assigned to these keys in each of the potentiostatic modes are displayed on the bottom line of the menu screen. These functions are described below. If something goes wrong and an error message is displayed, see Section 6.7 for an explanation.

F1 (Coulombs or offset I): This key selects the parameter to be displayed on the right side of the second line of the menu screen.

By pressing the F1 key, you can display either of two parameters on this line: net current with I OFFSET applied (i) or the final current integral (Q). I OFFSET is explained in Sections 6.3 and 6.4. The final current integral is the net charge, after the algebraic addition of I OFFSET, that has passed through the cell since the beginning of the experiment while the cell was on (or since the coulometer was reset). It is expressed in coulombs.

F2 (Q-Reset): The F2 key has the Q Reset (Q-Reset) function. When F2 is pressed, the following query is displayed on line 3 of the LCD panel:

RESET COULOMBS? . . .

If you press YES (F4), it resets the coulometer to zero and reestablishes the initial scaling factor, as set by the selected current range. If F1 has been used to display Q, you will see the final current integral (in coulombs) on the second line of the display reset to zero and then begin to increment as charge accumulation resumes.

If you press NO (F5), the coulometer is not reset and the previous menu screen reappears.

F3 (Start or Stop): The F3 key is used to start a scan. When it is pressed, the scan progresses as programmed. After the scan begins, the function assigned to F3 changes to "Stop". Pressing F3 ends the scan sequence and immediately reestablishes E INIT.

F4 (Hold or Resume): Pressing the F4 key during a scan causes the scan to temporarily halt. When the key is

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pressed again (it will now be labeled "Resume"), the scan resumes.

If a "Hold" occurs when a potential is applied, it will continue to be applied (assuming that the CELL switch is on). This is true if the hold takes place at any time during the scan sequence. The scan simply halts and the potential in effect at the moment of the halt continues to be applied. Time is not counted for the duration of the hold.

If F2 (**Q-Reset**) is pressed to reset the coulomb count, a prompt appears asking whether you want to proceed. To provide a way to respond to the query, F4 is temporarily reassigned the YES function.

F5 (Reverse or Bypass): In the Potentiostat Scan mode, the F5 key is assigned the "Reverse" function. Pressing F5 during a scan reverses the direction of the scan.

In the Potentiostat Step mode, the F5 key has a different function: Bypass. Pressing Bypass before or during the programmed T INIT, T MID, or T FINAL delay in the scanning sequence temporarily overrides that delay and sets it to a minimum (actually 4 ms). The scanning sequence then immediately advances to the next step. For example, if F5 is pressed during the T INIT interval, the sequence would immediately advance to the first scan. In half-cycle or single-cycle scanning, if the next step would advance the sequence to the end of the scan, the scan would halt. For an explanation of the T INIT, T MID, and T FINAL delays, see Chapter 8.

If F2 (Q-Reset) is pressed to reset the coulomb count, a prompt appears asking whether you want to proceed. To provide a way to respond to the query, F5 is temporarily reassigned the NO function.

6.7 ERROR MESSAGES

Certain conditions that interfere with normal operation will cause error messages to be displayed on the LCD panel. The message will give a warning statement about the type of error, information about the cause (sometimes in a numeric code), and usually advice about what action to take. Chapter 5, Section 5.10 shows the error messages that could be displayed, explains their meanings, and describes the actions you should take in response to each.

Another class of error messages can occur in remote-control operation as a result of problems in setting up or running software on the host computer. These error messages appear on the computer monitor screen, not the LCD panel. For a discussion of these messages, refer to the ERR command in the *Model 263A Command Set Handbook*, Chapter 2, Section 2.13. Please note that errors indicated on the LCD panel are hardware errors and are not related to the software error messages that you can display on the computer monitor with the ERR command.

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Chapter 7
Running Galvanostatic Experiments

7.1 INTRODUCTION

This chapter shows you how to set the experiment parameters that are unique to galvanostatic operation, and how to run your galvanostatic experiments.

But before you proceed any further, it is important to become familiar with the information in some of the previous chapters. Chapter 2 describes important safety precautions for operating this instrument. Chapter 3 shows you how to connect the instrument to your electrochemical cell and any other equipment you may be using with it. Chapter 4 takes you through a sample session with the instrument to introduce you to its operation.

Chapter 5 is particularly important, as it describes the functions of the controls and indicator lights on the front panel. It also shows you how to move among the eight menu screens that are available on the LCD display. It then explains how to set up the basic experiment parameters that seldom require changing. (These are set on the [System Default] and [System Interface] menus.)

Chapter 5 also shows how to set the communications parameters necessary for controlling the instrument from a host computer. Instructions for operating the unit remotely via either the RS-232 or GPIB (IEEE-488) interface port are provided in the separately bound *Model 263A Command Set Handbook*.

7.2 GALVANOSTATIC MODES

Under front-panel control, the Model 263A can hold the cell current fixed at a single level during the experiment, or it can vary the current between three separate programmable levels, called I Init, I Mid, and I Final.

The transition between the three levels can be in the form of either a gradual ramp or "scan" between levels, or a sudden sharp "step." So there are actually a total of three modes of galvanostatic operation available:

- Galvanostatic with constant, nonvarying cell current [Galvanostat].
- Galvanostatic Scan [Galvanostatic Scan], with a gradual ramp between the three levels of cell current. You can program the rate of change ("SCANRATE") of the current between levels.
- Galvanostatic Step [Galvanostatic Step], with sharp steps between the three levels of cell current.

The lengths of time for which each of the three levels of current are held constant during the experiment are called T Init, T Mid, and T Final. Each time can be separately programmed (or omitted entirely).

To run a galvanostatic experiment from the front panel, first set up the basic parameters as described in Chapter 5. Then, press the MODE key as many times as necessary to display the menu screen for the galvanostatic mode you wish to use: [Galvanostat], [Galvanostatic Scan], or [Galvanostatic Step].

After you select the appropriate menu screen, use the PREV and NEXT PARAMETER keys to display each experiment parameter in turn, as described in Chapter 5. The parameters used in each galvanostatic mode are described in the following sections. More detailed explanations are given in Chapter 8. If the default value of any parameter is not satisfactory for your experiment, use the VALUE knob to change it.

7.3 SETTING CELL CURRENT: SPECIAL CONSIDERATIONS

It is important to understand that if you change the full-scale current range, the instrument will automatically reset the cell current to maintain the same ratio of current to full-scale current range. For example, let's say the full-scale current range is 10 mA and cell current is set to 8.00 mA. Then cell current will be 80% of the full-scale current range. If you decrease the full-scale current range to 1 mA, this ratio will be maintained automatically and cell current will be reset to 0.800 mA. This is true for any current parameter—CURRENT, I INIT, I MID, and I FINAL.

Note that the value specified for any cell current parameter can be no greater than two times the full-scale current range selected.

7.4 SETTING CELL CURRENT IN A FIXED-CURRENT EXPERIMENT

The [Galvanostat] menu allows you to set the amount of cell current used in steady-state galvanostatic operation, where the cell current is held constant. The value for the CURRENT parameter is that in effect at the time the instrument was last powered down, or the last value set if changed since power-up. After you have set it as required for your experiment, follow the directions in Section 7.7 for running your experiment.

7.5 SETTING SCANNED-CURRENT EXPERIMENT PARAMETERS

The [Galvanostatic Scan] menu allows you to set the following experiment parameters for the Galvanostat Scan mode. In this mode, the Model 263A varies the cell current in a gradual sweep or ramp between steady-state levels.

- I INIT, the cell current during the first steady-state stage of a scan or step sequence.

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- I MID, the cell current during the second steady-state stage (sometimes called the "vertex") of a scan or step sequence.

- I FINAL, the cell current during the third steady-state stage of a scan or step sequence.

- T INIT, the length of time that I INIT is maintained in the cell.

- T MID, the length of time that I MID is maintained in the cell.

- T FINAL, the length of time that I FINAL is maintained in the cell.

- SCANRATE, the rate of change of the cell current between I INIT and I MID, and between I MID and I FINAL.

- CYCLES allows selection of three different scan or step sequences: HALF CYCLE, FULL CYCLE, and CONTINUOUS. The cycle setting determines the number of separate steady-state cell current levels that will be used during your experiment.

- CELL@END determines the cell currrent at the end of a scan or step cycle. If you select OFF, the cell will be turned off (counter electrode connection interrupted) at the end of the programmed cycle. If you select I INIT, the cell current will be returned (stepped sharply) to I INIT.

  If you select I FINAL, the cell currrent remains at its last value in the sequence. In the FULL CYCLE mode, this is I FINAL. But note that in HALF CYCLE operation, the last current level reached in the sequence actually is I MID.

  Also note that in the CONTINUOUS cycle mode, the experiment doesn't end until the "Stop" key (F3) is pressed. At that point, the cell current returns to I INIT.

These parameters are explained in greater detail in Chapter 8. After you have set them as required for your experiment, follow the directions in Section 7.7 for running your experiment.

7.6 SETTING STEPPED-CURRENT EXPERIMENT PARAMETERS

In the Galvanostat Step mode, the Model 263A varies the cell current in sharp steps between steady-state levels. The experiment parameters that can be set from the [Galvanostatic Step] menu are the same as those set from the [Galvanostatic Scan] menu (see above), except that there is no SCANRATE setting. After you have set the parameters as required for your experiment, follow the directions in Section 7.7 for running your experiment.

7.7 RUNNING YOUR EXPERIMENT

After you have connected your equipment (as described in Chapter 3) and set all required parameters (as described in Chapter 5 and this chapter), you are ready to begin your experiment. You will start the experiment and control its progress with the "soft" function keys, F1 through F5, below the LCD panel.

The functions assigned to these keys in each of the galvanostatic modes are displayed on the bottom line of the menu screen. These functions are described below. If something goes wrong and an error message is displayed, see Section 7.8 for an explanation.

F1 (Coulomb or offset I): This key selects the parameter to be displayed on the right side of the second line of the menu screen.

By pressing the F1 key, you can display either of two other parameters on this line: net current with current offset applied (i) or the final current integral (Q).

Current offset ("I OFFSET") is a feature that can be used in potentiostatic experiments but is not useful in galvanostatic operation. Thus there is no provision for setting it from the galvanostatic menus. But be aware that if you have set a value for I OFFSET in one of the potentiostatic menus and then switch to galvanostatic operation, the offset will remain in effect when "i" is displayed.

I OFFSET is a current value that is mathematically subtracted from the cell current before it is displayed. The result of this subtraction is displayed as "i" and accumulated over time as "Q". That is, i = I - (I OFFSET), where I OFFSET can have a positive or negative value.

Note that there is no actual current subtraction, only a numerical calculation. Thus current offset does not affect the potential at the I OUTPUT connector.

The final current integral (Q) is the net charge, after the algebraic addition of I OFFSET, that has passed through the cell since the beginning of the experiment while the cell was on (or since the coulometer was reset). It is expressed in coulombs.

F2 (Q-Reset): When F2 is pressed, the following query is displayed on line 3 of the LCD panel:

RESET COULOMBS? . . .

If you press YES (F4), it resets the coulometer to zero and reestablishes the initial scaling factor, as set by the selected current range. If F1 has been used to display Q, you will see the final current integral (in coulombs) on the second line of the display reset to zero and then begin to increment as charge accumulation resumes.

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If you press NO (F5), the coulometer is not reset and the previous menu screen reappears.

F3 (Start or Stop): The F3 key is used to start a scan. When it is pressed, the scan progresses as programmed. After the scan begins, the function assigned to F3 changes to "Stop".Pressing F3 ends the scan sequence and immediately reestablishes I INIT.

F4 (Hold or Resume): Pressing the F4 key during a scan causes the scan to temporarily halt. When the key is pressed again (it will now be labeled "Resume"), the scan resumes.

If a "Hold" occurs when a current is applied to the working electrode, it will continue to be applied (assuming that the CELL switch is on). This is true if the hold takes place at any time during the scan sequence. The scan simply halts and the current in effect at the moment of the halt continues to be applied. Time is not counted for the duration of the hold.

If F2 (Q-Reset) is pressed to reset the coulomb count, a prompt appears asking whether you want to proceed. To provide a way to respond to the query, F4 is temporarily reassigned the YES function.

F5 (Reverse or Bypass): In the Galvanostat Scan mode, the F5 key has the "Reverse" function. Pressing F5 during a scan reverses the direction of the scan.

In the Galvanostat Step mode, the F5 key has a different function: Bypass. Pressing F5 before or during the programmed T INIT, T MID, or T FINAL delay in the scanning sequence temporarily overrides that delay and sets it to a minimum (actually 4 ms). The scanning sequence then immediately advances to the next step. For example, if F5 is pressed during the T INIT interval, the sequence would immediately advance to the first scan. In half-cycle or single-cycle scanning, if the next step would advance the sequence to the end of the scan, the scan would halt. For an explanation of the T INIT, T MID, and T FINAL delays, see Chapter 8.

If F2 (Q-Reset) is pressed to reset the coulomb count, a prompt appears asking whether you want to proceed. To provide a way to respond to the query, F5 is temporarily reassigned the NO function.

7.8 ERROR MESSAGES

Certain conditions that interfere with normal operation will cause error messages to be displayed on the LCD panel. The message will give a warning statement about the type of error, information about the cause (sometimes in a numeric code), and usually advice about what action to take. Chapter 5, Section 5.10 shows the error messages that could be displayed, explains their meanings, and describes the actions you should take in response to each.

Please note that errors indicated on the LCD panel are hardware errors and are not related to the software error codes that you can display on the computer monitor with the ERR command.

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Chapter 8
Experiment Parameters

8.1 INTRODUCTION

This chapter is an alphabetical list of all the experiment setup parameters and commands that can be programmed from the front panel of the Model 263A, with detailed explanations of each. Refer to Chapters 5, 6, and 7 for information on how to set the parameters.

8.2 FRONT-PANEL PARAMETERS AND COMMANDS

Address
*See **GPIB Address**.*

Automatic Current Ranging
*See **Current Auto-Ranging; Current Range**.*

Bandwidth (Stability versus Speed)
Bandwidth is set from the FUNCTION menu screen. One of the two bandwidth modes, HI SPEED or HI STABILITY, will always be in effect. The default setting is HI STABILITY.

The HI SPEED mode gives the fastest response in Potentiostat or Galvanostat operation, but without guaranteed stability in all experiments. Under the proper conditions with certain cells, ringing almost always occurs and oscillations may occur. Cells having large capacitance and low resistance are most likely to prove troublesome.

The HI STABILITY mode provides extreme stability at somewhat reduced speed. In this mode, stable operation is assured with virtually any cell. However, the possibility always exists that with an unfamiliar cell configuration, or under extremely unusual operating conditions, stability problems might occur. If oscillation does occur in the HI STABILITY mode, the cause may be too much resistance in the reference electrode. Even a well-designed, low-impedance reference electrode can develop a high resistance if salts precipitate in the junction.

Baud Rate
*See **Serial Baud Rate**.*

Cell (Condition) at End
This parameter, set from the scan or step experiment menu screens, determines the working electrode potential (in potentiostatic operation) or cell current (galvanostatic operation) at the end of a scan or step cycle. If you select OFF (the default setting), the cell will be turned off (counter electrode connection interrupted) at the end of the programmed cycle. If you select E INIT (I INIT in galvanostatic operation), the working electrode potential (or cell current) will be returned to E INIT (or I INIT).

If you select E FINAL (or I FINAL), the working electrode potential (or cell current) remains at its last value in the programmed scan sequence. In the FULL CYCLE mode, this is E FINAL (or I FINAL). But note that in HALF CYCLE operation, the last level reached in the sequence actually is E MID (or I MID).

Also note that in the CONTINUOUS cycle mode, the experiment doesn't end until the "Stop" key (F3) is pressed. At that point, the cell status returns to E INIT (I INIT in galvanostatic operation).

Cell Current
Cell current is the controlled parameter in galvanostatic operation. In steady-state galvanostatic operation, where the cell current is held constant, the CURRENT parameter is set from the [Galvanostat] menu screen. The value for CURRENT is that in effect at the time the instrument was last powered down, or the last value set if changed since power-up. The units and resolution are indicated by the display.

Current parameters I INIT, I MID, and I FINAL, set from the [Galvanostatic Scan] and [Galvanostatic Step] menu screens, specify the cell current at each of three stages in a scan or pulse sequence. I INIT specifies the current at the start of the scan. I MID specifies the current at end of the scan's first leg. In Half Cycle mode operation, the scan ends at I MID, and I MID continues to flow in the cell.

In Full Cycle operation, I MID is a vertex and I FINAL specifies the final current which continues to flow after the scan is over. In Continuous Cycle operation, there is repetitive cycling between I MID and I FINAL until the STOP key (F3) is pressed. Delays T INIT and T MID may be used to determine the length of time I INIT and I MID are in effect (PASS = 4 ms). The Delay at I INIT and I MID may be carried out with the cell either on or off. See **Delays** for details.

It is important to understand that if you change the full-scale current range, the instrument will automatically reset the cell current to maintain the same ratio of current to full-scale current range. For example, let's say the full-scale current range is 10 mA and cell current is set to 8.00 mA. Then cell current will be 80% of the full-scale current range. If you decrease the full-scale current range to 1 mA, this ratio will be maintained automatically and cell current will be reset to 0.800 mA. This is true for any current parameter—CURRENT, I INIT, I MID, and I FINAL.

Note that the value specified for any cell current parameter can be no greater than two times the full-scale current range selected.

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Cell Potential
E INIT, E MID, and E FINAL specify the potential applied at each of three stages in a scan or pulse sequence. Potential is the controlled parameter in Potentiostat mode operation. E INIT specifies the potential applied at the start of the scan. E MID specifies the potential applied at end of the scan's first leg. In Half Cycle mode operation, the scan ends at E MID, and E MID continues to be applied.

In Full Cycle operation, E MID is a vertex and E FINAL specifies the final potential, which continues to be applied after the scan is over. In Continuous Cycle operation, there is repetitive cycling between E MID and E FINAL until the "Stop" key (F3) is pressed. Delays T INIT and T MID may be used to determine the length of time E INIT and E MID are applied (PASS = 4 ms). The Delay at E INIT and E MID may be carried out with the cell either on or off. See **Delays** for details.

In steady-state potentiostatic operation, where the potential at the working electrode is held constant with respect to the reference electrode, the Potential parameter is set from the [Potentiostat] menu screen. In steady-state operation, the value for the Potential parameter is that in effect at the time the instrument was last powered down, or the last value set if changed since power-up.

The units and resolution are indicated by the display. Potential can be specified to the nearest mV. Potentials can be set over a range of -10 V to +10 V. However, no given scan can span more than 4 V absolute. In other words, the difference between the highest potential and the lowest potential cannot exceed 4 V. *This range is extended with the 93 Option.*

Cell Type (REAL or DUMMY)
Set from the [System Default] menu screen, this parameter allows you to substitute an internal 10 kΩ, 0.01% resistor for a connected cell. This dummy load is useful for determining that the Model 263A is functioning correctly. Use REAL for normal operation with a cell.

Current, Cell
*See **Cell Current**.*

Current Auto-Ranging
In potentiostatic operation, you can allow the Model 263A to automatically set the appropriate full-scale current range. In the Auto-Ranging mode, selected by pressing the AUTO key, the Model 263A automatically seeks the full-scale current range that causes the I OUTPUT potential to be between 15% and 190% of full scale; that is, between 150 mV and 1.9 V. If cell current is less than 150 nA, the 100 nA range will be selected. If the instrument is on the 10 mA range and cell current is greater than 19 mA, the 100 mA range will be selected. This process occurs after each point is acquired, with the constraint that it can only change one range at a time. Data could be lost if the data amplitude changed faster than it could be tracked by the auto-ranging function. For an explanation of the full-scale current range, see **Current Range, Full Scale**.

You can set a limit to the automatic current ranging by adjusting the I AUTO LIMIT parameter on the [System Default] menu (see Chapter 5, Section 5.6). The full-scale current range will not go below the I AUTO LIMIT you set. The default range is 100 nA.

Note that automatic current ranging is restricted to operation in the Potentiostat mode. It cannot be selected in the Galvanostat mode.

Current Integral (Coulombs) Display
In the potentiostatic and galvanostatic experiment menu screens, the F1 key selects the parameter to be displayed on the right side of the second line of the menu screen.

By pressing F1, you can display either of two other parameters on this line: net current with I OFFSET applied (i) or the final current integral (Q).

I OFFSET is a current value that is mathematically subtracted from the cell current before it is displayed. The result of this subtraction is displayed as "i" and accumulated over time as "Q". That is, i = I - (I OFFSET), where I OFFSET can have a positive or negative value.

Note that there is no actual current subtraction, only a numerical calculation. Thus current offset does not affect the potential at the I OUTPUT connector.

Current offset is a feature that can be used in potentiostatic experiments but is not useful in galvanostatic operation. Thus there is no provision for setting it from the galvanostatic menus. But be aware that if you have set a value for I OFFSET in one of the potentiostatic menus and then switch to galvanostatic operation, the offset will remain in effect when "i" is displayed.

The final current integral (Q) is the net charge, after the algebraic addition of I OFFSET, that has passed through the cell since the beginning of the experiment while the cell was on (or since the coulometer was reset). It is expressed in coulombs. In FULL FRILLS operation (available only in remote control from a host computer), the displayed charge accumulation will be updated every timebase. Q normally is not selected if speed of data acquisition is the main concern, such as might be the case when the Model 263A is being controlled from an external computer via the RS-232 or GPIB interface.

When you press the F2 key (Q Reset), the following query is displayed on line 3 of the LCD panel:

RESET COULOMBS? . . .

If you press YES (F4), it resets the coulometer to zero and reestablishes the initial scaling factor, as set by the

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selected current range. If F1 has been used to display Q, you will see the final current integral (in coulombs) on the second line of the display reset to zero and then begin to increment as charge accumulation resumes.

If you press NO (F5), the coulometer is not reset and the previous menu screen reappears.

In a perfect instrument and cell system, Q of course could not change when the cell is disconnected, because no cell current would flow. But in a real system, no matter how precise, some small charge may appear as a result of drift and other circuit variations. Thus, setting UPDATE Q W/CELL OFF = NO (from the [System Default] menu) prevents charge from accumulating when the cell is off. Choosing YES allows it to accumulate, and might help to estimate drift errors.

Current Interrupt iR Compensation

Current Interrupt is one of two available iR Compensation techniques (see **iR Compensation**). iR Compensation can be selected from any of the three potentiostatic menu screens (IR MODE). The default setting is OFF.

iR Compensation is used to eliminate the error caused by the iR drop across the uncompensated resistance in the electrochemical cell. The Current Interrupt technique does this by rapidly shutting off the cell current, estimating the drop in reference potential before the cell junction capacitance discharges through R_p (the polarization resistance), and then adding that iR drop back into the counter electrode control voltage.

When the current in the cell is interrupted, a drop in the E OUTPUT potential equal to iR_U occurs. As this potential drop cannot be measured directly, it is estimated by extrapolation from two other potential levels measured at specified intervals after current interruption.

Four parameters, all available from the potentiostatic menu screens, can be adjusted to estimate and fine-tune the amount of iR compensation to be applied. IRUPT (Intermittent Current Interrupt) sets the interval between current interrupts. The range is 1 to 32767 data points, with a default setting of 5 points. In local operation from the front panel, 5 points is equivalent to 20 ms if LINE SYNC is not enabled. (In remote control from a host computer, the interrupt timing is controlled by the computer.)

IRUPT T1 sets the increment of time between the interruption of current to the cell and the first sampling of potential during the no-current condition. IRUPT T2 sets the time increment from IRUPT T1 to the second sampling of potential. These two potential levels, at IRUPT T1 and IRUPT T2, are used to extrapolate the value of the iR drop across the uncompensated resistance in the cell.

The IRUPT T1 and IRUPT T2 settings can range from 10 μs to 2000 μs, but the sum of the values of IRUPT T1 and IRUPT T2 may not exceed 2000 μs. The default values for both are 10 μs on the 100 mA current range (and on the 1 A range if 94 option is installed) and 75 μs on all other current ranges. Note that IRUPT T1 and IRUPT T2 *can be set independently for each current range.*

IRPC (Current Interrupt Percent Correction) sets the percentage of correction, adjustable from 0% to 200%, with a default of 100%. This function is used to fine-tune the extrapolation procedure.

Note: The default values of these parameters give good results in many applications. Do not change them unless you are well versed in current-interrupt iR compensation theory. This is particularly true of IRUPT T1, IRUPT T2, and IRPC.

A prerequisite to evaluating these parameter selections is that an oscilloscope be connected to the E OUTPUT BNC connector to observe the current-interrupt waveform, as discussed in the following paragraphs. Even with the oscilloscope it is not easy to make optimum determinations. The position of the selected points, as well as the path of the projection through the points, must be estimated.

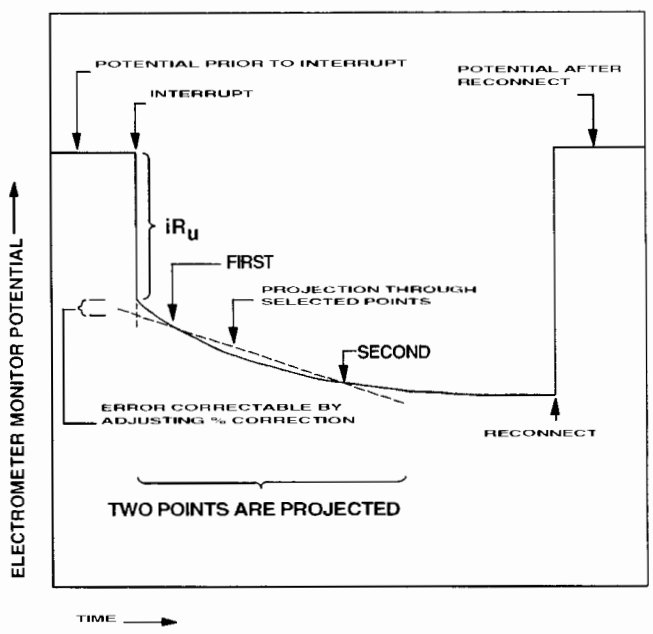


Figure 8-1. CURRENT INTERRUPT WAVEFORM AT E OUTPUT CONNECTOR

Figure 8-1 illustrates the E OUTPUT waveform for a typical Current Interrupt cycle. Referring to the figure, note that a fast potential drop equal to iR_U occurs at the moment of interrupt. The purpose of the current interrupt procedure is to estimate this potential drop so that R_U can be calculated and a correction factor developed. As the drop cannot be measured directly, it is estimated by

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extrapolation from two potential levels measured later at different points on the curve of potential decay after current interruption.

Just before current interruption, the potential level is sampled. After interrupt, two more potential samples (at IRUPT T1 and T2) are taken. The operator specifies the time intervals between the current interrupt and the second sample (IRUPT T1), and between IRUPT T1 and the third sample (IRUPT T2). Each time interval must be at least 10 μs.

The potential levels at IRUPT T1 and T2 are used to define a projection that is extrapolated back to the instant just after current interruption. By comparing the linearly extrapolated potential at that instant with that before the interruption (the first sample), a correction function is developed that corrects for the uncompensated iR drop in the cell.

The goal is to select two points for IRUPT T1 and T2 that will provide a projection that intersects the lower end of the iR_U drop. If the intersection occurs at a higher point, the correction factor will be too small. If it occurs at a lower point, as is the case for the example depicted in the figure, the correction factor will be too large.

Two general guidelines can be used for setting IRUPT T1 and IRUPT T2:

1. T1 should be greater than cell cable capacitance (about 20 pf) $\times$ R_U (often on the order of 1000 Ω). Assuming these values, IRUPT T1 should be greater than 20 ns. But R_U may be considerably higher than 1000 Ω when you are working with low-conductivity media, such as concrete or nonaqueous solvents.

2. T2 should be less than cell capacitance (generally around 25 μF/cm^2) $\times$ R_P (as determined from a polarization resistance experiment).

Fortunately, IRPC, by allowing user control of the percentage of correction, eases the point selection task. For example, if the extrapolated line were to intersect exactly halfway down the drop, the correction factor would be 50% of the required value. The resulting correction factor could be easily adjusted to its proper value by using IRPC to set the percentage of correction to 200%. Similarly, if the intersection occurred below the drop, as in the depicted example, IRPC would be used to set a percentage of correction of less than 100% (90% would be about right for the example).

Once Current Interrupt iR Compensation is established by setting IR MODE = IRUPT, and IRUPT T1, IRUPT T2, IRUPT, and IRPC are set, no other operator intervention is required during the measurement.

Current Interrupt iR Compensation has several advantages as compared to Positive Feedback iR Compensation:

1. This technique corrects for essentially the entire potential error caused by the uncompensated resistance.

2. This technique corrects for any changes in the uncompensated resistance as the scan progresses.

3. No adjustment is required on the part of the user. The only action required is the simple selection of IR MODE = IRUPT. (The default values of IRUPT T1, IRUPT T2, IRUPT, and IRPC will give good results in many applications.)

Disadvantages of the technique include:

1. The correction occurs at finite times. Thus the technique is not suitable for scan rates greater than 500 mV/s and may, in fact, not work properly at scan rates greater than 100 mV/s.

2. If the correction isn't updated frequently enough, the applied correction can be in error.

3. In some cases, this technique can cause the entire system to oscillate. Note that Current Interrupt is a positive feedback technique which reduces the margin of stability in the control of the cell. Slow scan rates reduce this problem by allowing the potentiostat loop to fully settle before the next interruption.

Note: If the cell is turned OFF while current interrupt is selected, the current interrupt routine is skipped and no correction occurs. When the cell is turned back on, the current-interrupt routine resumes.

Once Current Interrupt Compensation is running, the user may wish to monitor the waveform at the E OUTPUT connector with an oscilloscope to optimize the Current Interrupt parameters as established by IRUPT T1, IRUPT T2, IRUPT, and IRPC. The selection of the extrapolation points (T1 and T2) and the Percent Correction (IRPC) setting determine the accuracy of the applied correction. Note that the waveform depicted in Figure 8-1 is idealized; actually encountered waveforms may not be so easy to analyze.

Current Offset (I Offset)

Available at the three potentiostatic operation menus, I OFFSET allows you to specify a current value to be mathematically subtracted from the cell current before it is displayed. The result of this subtraction is displayed as "i" and accumulated over time as "Q". That is, i = I - (I OFFSET), where I OFFSET can have a positive or negative value.

Note that there is no actual current subtraction, only a numerical calculation. Thus current offset does not affect the potential at the I OUTPUT connector.

The offset range is ± 2 times the selected full-scale current range. If you change the current range, the instrument will

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automatically reset I OFFSET to maintain the same ratio of currrent offset to full-scale current range.

Current Overload
The I OVERLOAD indicator lights if the cell current exceeds two times the selected full-scale current range. This does not mean that the cell control loop is out of control. It means only that the cell current monitoring circuits are being driven to the limit.

Current Range, Full Scale
The full-scale current range selection is used to scale the output potential at the I OUTPUT connector to a useful value for any cell current. A cell current equal to the selected current range gives 1 V at the I OUTPUT connector. Note that a current that exceeds two times the current range generates an I OVERLOAD condition.

The polarity of the voltage at the I OUTPUT connector is that of the current. For example, a current level of -1 mA will give -1 V at the I OUTPUT connector (1 mA Current Range), representing an anodic or reducing current.

The full-scale current range can be set either manually by the operator or automatically by the Model 263A ("Auto-Ranging"). But note that Auto-Ranging is restricted to potentiostatic operation. Should the user attempt to select Auto-Ranging in galvanostatic operation under remote control, an error message will be generated. In local control, no error message will be generated but the AUTO key will be ignored.

In the Manual ranging mode, selected by toggling the AUTO key to turn the AUTO indicator off, the current range is selected with the arrow keys below the current range indicators. The ↑ ↑ key moves the selection upwards through the available ranges. The ↓ ↓ key moves it downwards.

In the AUTO mode, selected by pressing the AUTO key, the Model 263A automatically seeks the full-scale current range that causes the I OUTPUT potential to be between 15% and 190% of full scale; that is, between 150 mV and 1.9 V. If cell current is less than 150 nA, the 100 nA range will be selected. If the instrument is on the 10 mA range and cell current is greater than 19 mA, the 100 mA range will be selected. This process occurs after each point is acquired, with the constraint that it can only change one range at a time. Data could be lost if the data amplitude changed faster than it could be tracked by the auto-ranging function.

You can set a limit to the automatic current ranging by adjusting the I AUTO LIMIT parameter on the [System Default] menu (see Chapter 5, Section 5.6). The full-scale current range will not go below the I AUTO LIMIT you set. The default range is 100 nA.

The I/E Filter (if selected) can cause unpredictable effects on your experiment if AUTO current ranging is in use. If I/E FILTER = ON is selected (from the [System Default] menu screen), you generally should use manual current ranging.

Current Ranging, Automatic
*See **Current Auto-Ranging**.*

Cycle Control
The CYCLES parameter, set from the [Potentiostatic Scan], [Potentiostatic Step], [Galvanostatic Scan], or [Galvanostatic Step] menu, determines the number of separate steady-state potential levels that will be used at the working electrode (or steady-state cell current levels in galvanostatic operation), during your experiment. Three different cycle sequences are available: HALF CYCLE, FULL CYCLE, or CONTINUOUS. A brief description of each setting follows.

1. HALF CYCLE: This is the simplest cycle, involving only two potential or current settings. A half-cycle sequence proceeds as follows.

   a. Delay T INIT at E INIT or I INIT.

   b. Scan (or step) from E INIT/I INIT to E MID or I MID.

   c. Scan remains at E MID or I MID for T MID, then stops.

   At the end of the scan or step sequence, the setting of CELL@END on the menu will determine whether cell potential or current will remain at its last value (CELL@END = E FINAL or I FINAL), will step to E INIT or I INIT, or will step to OFF (counter electrode connection interrupted).

   Note that the setting of CELL@END = E FINAL or I FINAL will cause cell potential or current to remain at the last level it reached in the experiment sequence, which in half-cycle operation actually is E MID or I MID.

2. FULL CYCLE: A Full Cycle is more complex and involves all three programmed potential or current levels. The sequence is:

   a. Delay T INIT at E INIT or I INIT.

   b. Scan (or step) from E INIT/I INIT to E MID or I MID.

   c. Delay T MID at E MID or I MID.

   d. Scan (or step) from E MID/I MID to E FINAL or I FINAL.

   e. Scan remains at E FINAL or I FINAL for T FINAL, then stops.

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At the end of the scan or step sequence, the setting of CELL@END on the menu will determine whether cell potential or current will remain at E FINAL or I FINAL, will step to E INIT or I INIT, or will step to OFF (counter electrode connection interrupted).

3. CONTINUOUS: Continuous is the most complex of the three cycles. The sequence is as follows.

   a. Delay T INIT at E INIT or I INIT.

   b. Scan (or step) from E INIT/I INIT to E MID or I MID.

   c. Delay T MID at E MID or I MID.

   d. Scan (or step) from E MID/I MID to E FINAL or I FINAL.

   e. Delay T FINAL at E FINAL or I FINAL.

   f. Scan (or step) from E FINAL/I FINAL to E MID or I MID.

   g. Steps 3 through 6 repeat until 'Stop" key is pressed.

   Automatic cycling of the scan back and forth between E MID/I MID and E FINAL/I FINAL will continue until you press the "Stop" key (F3). When it is halted, the cell potential or current will step sharply to the E INIT/I INIT.

Data Bits
*See **Serial Data Bits**.*

Delays T Init, T Mid, T Final
A delay is a time interval between rampwise or stepwise changes in cell potential or current. The cell potential or current does not change during a delay. Delays are programmed from a "Scan" or "Step" menu, and can be set at three different points (T INIT, T MID, and T FINAL) in the scan or step sequence. Each delay can range from 4 ms to 500 s.

Scans typically, but not necessarily, begin with a delay. The first delay interval, T INIT, precedes the first leg of the scan or step. It begins when you press "Run" (F3). The cell potential or current during T INIT is determined by the setting of E INIT or I INIT. If E/I INIT is not zero, the cell potential or current steps sharply to the specified level. Once the delay interval ends, the scan begins and the potential or current begins to increment as programmed.

In full-cycle or continuous operation, a second delay, T MID, can be interposed between the first and second legs of the scan. The cell potential or current is determined by the setting of E MID or I MID.

In continuous operation only, a third delay, T FINAL, can be interposed between the second and third legs of the scan. The cell potential or current is determined by the setting of E FINAL or I FINAL.

In [Potentiostatic Step] or [Galvanostatic Step] operation, you may temporarily override a programmed T INIT, T MID, or T FINAL delay by pressing "Bypass" (F5). Pressing F5 before or during the delay temporarily overrides that delay and sets it to a minimum (actually 4 ms). The scanning sequence then immediately advances to the next step.

For example, if "Bypass" F5 is pressed during the T INIT interval, the sequence would immediately advance to the first scan. In half-cycle or single-cycle scanning, if the next step would advance the sequence to the end of the scan, the scan would halt.

Dummy Cell
*See **Cell Type**.*

E Initial, E Mid, E Final
*See **Cell Potential**.*

E Overload
The E OVERLOAD indicator lights if the control amplifier output is at its limit ($E_{control}$ > 20 V) and the control loop is not controlling the cell potential. This can happen in either Potentiostatic or Galvanostatic operation and normally indicates either a cell-setup problem or an extremely high resistance solution.

Whenever E OVERLOAD lights, the problem *must* be located and corrected before valid measurements can be made. A common cause in potentiostatic operation is a disconnected reference electrode. Note that this can cause damage to the working electrode if it occurs.

Echo
*See **GPIB Echo; Serial Echo**.*

External Input
The EXT INPUT connector has two separate but similar functions: External Input and Oscillator Input. The main difference between these functions is that in the OSC IN mode the gain of the applied signal can be scaled up or down, while the EXT INPUT mode always applies unity gain to the input signal.

This connector can be made active (turned on) or inactive either with the front panel controls or by software commands from an external computer. From the front panel, it can be controlled with either the EXT INPUT or the OSC IN setting on the [System Default] menu (see "Adding an External Signal" in Section 3.4). Remote control from an attached computer is described in the *Model 263A Command Set Handbook*. Note that both EXT INPUT and OSC IN cannot be set to ON at the same time.

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When the EXT INPUT connector is active ("ON"), an analog control voltage applied to it will be summed with the output of the digital-to-analog converters (DACs), which are set at the front panel or from an attached PC. The control signals could be generated by, for example, an EG&G PARC Model 175 Universal Programmer to provide rapid scanning and true analog cyclic voltammetry. Other waveform generators, a lock-in amplifier, or a frequency response analyzer could be used.

In potentiostatic operation, all of the control potentials add algebraically. For example, if the voltage applied to EXT INPUT is +0.5 V, at a time when a potential of +0.5 V is programmed in the instrument, the net potential applied will be +1.0 V, and the working electrode would be controlled at +1.0 V with respect to the reference electrode. (This assumes, of course, that the applied signal is not scaled by selecting the OSC IN mode.) A positive applied potential will make the current tend to be more anodic. A negative applied potential will make the current tend to be more cathodic.

In galvanostatic operation, making the applied potential more positive by any means except applying a potential to the EXT INPUT connector will tend to make the cell current more cathodic. Making the applied potential more negative will tend to make the current more anodic. *This sense is reversed at the EXT INPUT connector. There, making the input more positive will make the cell current more anodic. Making the input more negative will make the cell current more cathodic.*

Extrapolation Points
See ***Current Interrupt iR Compensation***.

Filters
You can switch in two low-pass filters by setting I/E FILTER = ON from the [System Default] menu screen. Note that you cannot select only one filter at a time; both must be either selected at the same time or bypassed. Both filter the signal input to the A/D converter. One of these filters has a sharp cutoff at 5.3 Hz. (In remote operation, a 590 Hz low-pass filter is also available under software control.)

The second low-pass filter selected by setting I/E FILTER = ON consists of a roll-off capacitor that reduces the bandwidth of the I/E converter. The effect of this capacitor on bandwidth depends on the current range selected.

Unlike the 5.3 Hz filter, the roll-off capacitor filters the signal at the I OUTPUT connector. This capacitor is often helpful when making low-current measurements on large electrodes because it reduces the noise at I OUTPUT. The disadvantage of using the filter is that the I OUTPUT response time is degraded.

Approximate cut-off frequencies for the roll-off filter capacitor at each current range, as measured at the I OUTPUT connector, are as follows:

	CUT-OFF FREQUENCY	
CURRENT RANGE	HI-STABILITY MODE	HI-SPEED MODE
100 mA	5.5 kHz	10.5 kHz
10 mA	5.5 kHz	9.7 kHz
1 mA	4.5 kHz	6.5 kHz
100 μA	1.2 kHz	1.3 kHz
10 μA	150 Hz	150 Hz
1 μA	15 Hz	15 Hz
100 nA	1.5 Hz	1.5 Hz

As the effect of the roll-off capacitor on the I/E converter bandwidth varies with the current range, filtering can cause unpredictable effects if AUTO current ranging is in use. If I/E FILTER = ON is selected, we recommend manual current ranging.

It may be advantageous to synchronize data acquisition with the power line frequency when filtering. This can be done by setting LINE SYNC = ON (see "Line Sync").

Full-Scale Current Range
See ***Current Range, Full Scale***.

GPIB Address
The IEEE 488-1978 Instrument Bus Standard defines a bit-parallel, byte-serial bus structure designed to allow communications between intelligent instruments. Using this standard, many instruments may be interconnected and remotely controlled or programmed. Data can be taken from, sent to, or transferred between instruments via one connector or port.

Each device on a shared GPIB bus is assigned a listen address and a talk address for identification. These addresses are set at the device. When the controller (the PC) wishes to communicate with a specific device (the Model 263A) on the bus, it places the listen address of the device on the bus. Only the device having the corresponding address will respond to the subsequent message. Similarly, if the controller wants a device to talk, it sends the talk address of the device in question. The addressed device will transmit until a different talker is designated.

When the controller communicates with a device on the bus, it begins by placing the address of that device on the bus with $\overline{\text{ATN}}$ asserted. Naturally, each device must "know" its own TALK and LISTEN address. In the case of the Model 263A, these addresses are set from the [System Interface] menu on the front panel with the VALUE knob.

The Model 263A can be set for any address from 1 to 30. The normal address setting for use with EG&G PARC software is 14. See your software manual for its individual requirements.

Further details of GPIB addressing are given in the appendixes of the *Model 263A Command Set Handbook*.

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GPIB Terminator Character
In GPIB communications, the terminator is the character that marks the end of each transmission from the Model 263A to the host computer or from the host computer to the Model 263A. It must be selected on the Model 263A according to the requirements of the host computer, sometimes in conjunction with the software it is running. Failure to satisfy this requirement is one of the most common causes of problems in establishing communications via the GPIB.

You set the terminator from the [System Interface] menu on the Model 263A for GPIB *transmissions* to the host computer. When the terminator is set to "CR", it is a Carriage Return. When the terminator is set to "CRLF", it is a Carriage Return followed by a Line Feed. The terminator required for GPIB communications with IBM PC-compatible computers running EG&G PARC software is CR.

Note that you cannot configure the Model 263A to *receive* transmissions ended by the CRLF terminator. The Model 263A always expects the CR terminator in data it is receiving via the GPIB bus.

GPIB Test Echo
A special GPIB TEST ECHO mode can be established by switching to the [System Interface] menu and setting GPIB ECHO = ON. When the Test Echo function is ON, every character transmitted or received via the GPIB port will be echoed to the RS-232 interface connector. The RS-232 baud rate should be high to prevent slowing down the GPIB.

This feature is particularly useful when developing programs. If a "dumb" CRT terminal is connected to the RS-232 interface, the programmer will see all communications on the CRT. Such a terminal will allow direct interaction, thereby facilitating the programmer's understanding of the commands and responses. In addition, the programmer will be able to monitor the program data and intervene if necessary.

During normal operation, GPIB ECHO should be set to OFF.

High-Speed Mode
*See **Bandwidth**.*

High-Stability Mode
*See **Bandwidth**.*

I Initial, I Mid, I Final
*See **Cell Current**.*

I Offset
*See **Current Offset**.*

I Overload
*See **Current Overload**.*

iR Compensation
In an electrochemical experiment, the voltage across the double layer of the specimen surface must be closely controlled. If you don't have confidence that the potential you're applying is the potential felt at the specimen, you can't be sure that your measurement is valid.

But there are factors which make it difficult to control this potential. In all electrochemical cells there is some resistance between the tip of the reference electrode and the outside of the double layer of the specimen surface. This resistance is referred to as the "uncompensated resistance."

As current flows through the electrochemical cell, a potential is developed across the uncompensated resistance. When the uncompensated resistance gets high (as it does in pure water or organic solvents, for example) or the current becomes large, a significant potential error may occur.

Obviously, it is desirable to eliminate this error. The Model 263A provides two techniques for this: Positive Feedback iR Compensation (PFIR) and Current Interrupt iR Compensation (IRUPT). The voltage at the E OUTPUT connector is corrected when either technique is employed.

Both iR Compensation techniques can be set from any of the three potentiostatic menu screens. Only one iR Compensation mode can be active at a time. If one of the two is active and the other is selected, the one previously active will be turned off and the newly selected mode will become active.

The two iR Compensation techniques are performed in entirely different ways. Because each of the techniques has strengths and weaknesses, some situations will call for one technique and some for the other. Table 8-1 briefly lists the relative strengths of the two techniques.

PARAMETER	POS. FEEDBACK	CURRENT INTERRUPT
SPEED	FAST	SLOW
EASE OF USE	MORE DIFFICULT	VERY SIMPLE
R_u TRACKING	DOES NOT TRACK	TRACKS
STABILITY	CRITICAL	VERY STABLE
ARTIFACTS	NONE	MINOR

Table 8-1. COMPARISON OF POSITIVE FEEDBACK AND CURRENT INTERRUPT iR COMPENSATION

For details of the two techniques, see **Current Interrupt iR Compensation**; **Positive Feedback iR Compensation**. A more complete discussion of iR Compensation can be found in "Technical Note 101,"available on request from EG&G Princeton Applied Research.

iR Mode
*See **iR Compensation**; **Current Interrupt iR Compensation**; **Positive Feedback iR Compensation***

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IRUPT
*See **Current Interrupt iR Compensation**.*

Knob Timeout
To prevent someone from changing an experiment parameter by inadvertently moving the VALUE knob, you can disable the knob after you have finished using it. You can specify the period of time it waits after its last use before becoming inactive (the "timeout" period). At the end of the timeout period the knob will have no effect on parameter settings, and the LCD panel will display "KNOB TIMEOUT HIT ANY KEY." Press any key to reactivate the knob. The KNOB TIMEOUT parameter is set from the [System Interface] menu screen.

Line Sync
The LINE SYNC parameter, set from the [System Default] menu screen, determines whether data acquisition will be synchronized with the power line frequency. If you set it to YES, samples are taken every 16.6 ms (60 Hz power) or every 20 ms (50 Hz power). If you set it to NO, samples are taken at the rate determined by the internal time base. The default state of this parameter is NO.

If the LINE SYNC setting is changed while a scan is in progress, the scan will proceed at the new rate.

Note: In P-Scan, if the **scan rate** is >250mV/s, Line Sync ***cannot*** be enabled. Conversly, if Line Sync is enabled, the **scan rate** will not increase beyond 250 mV/s. A similar interaction applies to G-Scan, where Line Sync is incompatible with scans faster than 25% of f.s. range per second. For example, on the 100 μA range, the fastest scan rate possible with Line Sync enabled would be 25 μA per second.

Osc In
Set from the [System Default] menu screen, the OSC IN parameter setting determines whether the front-panel EXT INPUT connector will accept and scale an applied signal. Use ON if the applied signal is too large or too small and you wish to scale it by a factor of 0.02, 0.2, or 2.0. OFF is used either when no signal is to be applied, or when an applied signal is of a useful amplitude with unity gain (in that case, use EXT INPUT = ON).

Note that both EXT INPUT and OSC IN cannot be set to ON at the same time. If you are unsuccessful in turning one on, check that the other is off.

If you set OSC IN = ON, press the NEXT PARAMETER key once and set OSC GAIN (see below).

Osc Gain
When the OSC IN parameter is set to ON, an external signal applied to the EXT INPUT connector will be scaled by a factor specified by the OSC GAIN parameter. Both parameters are set from the [System Default] menu screen. The provided scaling factors are: 0.02, 0.2, and 2.

Parity
*See **Serial Parity**.*

Positive Feedback iR Compensation (PFIR)
Positive Feedback is one of two available iR Compensation techniques (see **iR Compensation**). iR Compensation can be selected from the any of the three potentiostatic menu screens (IR MODE). The default setting is OFF.

In the Positive Feedback technique, an analog positive feedback loop is established from the output of the current measurement circuit to the Control Amplifier input. The user sets the loop gain to approximately the value required to compensate for R_U.

The principal advantage of Positive Feedback is that the correction is continuous. As a result, correction remains effective even at the fastest scans possible with the Model 263A. While this technique is the only one possible for rapid scans (scans of 100 mV/s or faster), there are several significant disadvantages.

1. Adjusting the feedback is a tedious and subjective process.

2. The feedback reduces the stability of the potentiostat and can lead to severe ringing or oscillation.

3. Because of the effect on system stability, it is generally not possible to achieve 100% compensation. Instead the user normally has to be satisfied with a correction in the range of 75% to 90%.

4. The feedback assumes that the uncompensated resistance is constant. Should it vary, the applied correction will be incorrect. R_U is frequently not stable; it can change with the applied potential, time, changes in cell geometry, and other factors.

Before the Positive Feedback Mode can be activated, it is necessary to set and enter the correct value for R_U, the value to be compensated. If the entered value is too low, R_U will not be adequately compensated. If it is too high, the system will be unstable and may even oscillate. Once R_U is entered, Positive Feedback iR Compensation can be activated. The resistance resolution limit and maximum compensation as a function of Current Range is:

CURRENT RANGE	MAXIMUM COMPENSATION	RESOLUTION LIMIT
100 nA	20 MΩ	10 kΩ
1 μA	2 MΩ	1 kΩ
10 μA	200 kΩ	100 Ω
100 μA	20 kΩ	10 Ω
1 mA	2 kΩ	1 Ω
10 mA	200 Ω	100 mΩ
100 mA	20 Ω	10 mΩ
1 A	2 Ω	1 mΩ

Note: 1 A range is only available if 94 option is installed.

Resistance values have a resolution limited to 0.05% of

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the current-measuring resistor associated with the Current Range. Thus, the accuracy of the value that is actually programmed can vary with the Current Range. Moreover, as a result of this resolution limit, the value in effect can change during the experiment if the Current Range changes either manually or automatically. For this reason, care should be taken when using Positive Feedback iR Compensation with auto-ranging.

Consider how the resolution limit affects the accuracy of the programmed value. For example, assume a resistance of 1.234kΩ is entered on the 100 μA range. On that range the resistance resolution is 10 Ω, giving an actual programmed resistance of 1230 Ω. In other words, the programmed resistance differs from that entered (front or rear panels) by 4 Ω.

Let us continue with this example to see how error due to the resolution limit can occur when the current range changes. If, during the experiment, the current range changes to 1 mA, where the resistance resolution is 1 Ω, the actual programmed resistance will change to 1234 Ω. The improved resolution allows the actual programmed value to be identically that originally entered. Although this *change* could be a problem in some situations, it will usually be relatively minor.

The real problem occurs when shifting to a more sensitive range. For instance, if, in the example, the current range were to shift to 10 μA, where the resolution limit is 100 Ω, the actual programmed resistance will become 1200 Ω because the value can only be represented to the nearest 100 Ω. In other words, the error will now total 34 Ω. Similarly, a further shift to 1 μA would give a resistance of 1000 Ω (234 Ω error) and a shift to 100 nA would give a resistance of 0 Ω (1234 Ω error). Although this is a fairly large error, it is generally not significant because the current is small.

Note: Should one later shift to less sensitive ranges, the error will be successively reduced with each current range step.

Clearly, operators need to be mindful of the resolution limit for the "setup" current range and should set a resistance appropriate to that limit. Also, if more sensitive ranges are used during the experiment, users will have to be mindful of the impact the changing resolution will have on the programmed resistance and of the possible consequences of large resistance errors.

Two procedures for establishing positive feedback compensation are provided. The first procedure applies when the value of R_U is known. The second applies when R_U is not known.

If R_U is known:

1. Set IR MODE = PFIR.
2. Press the NEXT PARAMETER key to display the PFIR COMP parameter. The current value of assumed R_U will be displayed.
3. Use the VALUE knob to set the value of R_U to be compensated.

If R_U is not known:

1. Set IR MODE = PFIR.
2. Press the NEXT PARAMETER key to display the PFIR COMP parameter. The current value of assumed R_U will be displayed.
3. Use the VALUE knob to set the value of R_U to 0.00 ohms.

   It is always advisable to start with zero iR compensation. With a higher starting value, there is a definite risk factor in that the system may oscillate when the Positive Feedback Mode is activated. High power, high amplitude oscillations can be destructive and definitely should be avoided.
4. Program the Model 263A to apply a 62.5 Hz, ±50 mV square wave to the cell. (The ±50 mV figure is not critical. Smaller or larger amplitudes, if more appropriate to the chemistry, can be used.) The following parameter values can be used.

   Note: If LINE SYNC is set to ON, the frequency will be 15 Hz (60 Hz power) or 12.5 Hz (50 Hz power). The procedure will generally be easier to perform with the LINE SYNC set to OFF (less flicker on the oscilloscope display).

MODE =	POTENTIOSTATIC STEP
E INIT =	-0.05 V with respect to the potential of interest
T INIT =	PASS (see Note below)
E MID =	+0.05 V with respect to the potential of interest
T MID =	PASS (see Note below)
E FINAL =	-0.05 V with respect to the potential of interest
CYCLES =	CONTINUOUS

   Note: 62.5 Hz rate results from setting both delays to PASS.
5. Set up the cell for the measurements to be made. (Any change at the cell will probably change R_U.)

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6. Set the full-scale current range to the range that will be used in the measurement. If the current range setting is changed, the actual value of iR compensation can change as previously discussed.

7. Press the CELL switch to establish the Cell ON state. Then press "Run" (F3) to apply the square wave to the cell.

8. Monitor the I OUTPUT connector with an oscilloscope. At the oscilloscope, use internal triggering and display the waveform so its rise time can be readily observed. A horizontal deflection factor of 2 ms per division will generally give good results.

9. Make sure the PFIR COMP parameter is shown on the LCD panel. Then, while observing the displayed waveform on the oscilloscope, turn the VALUE knob slowly clockwise. The R_U compensation will increase, causing the decay intervals in the displayed waveform to become shorter. You can see the actual value of R_U compensation changing on the Model 263A display.

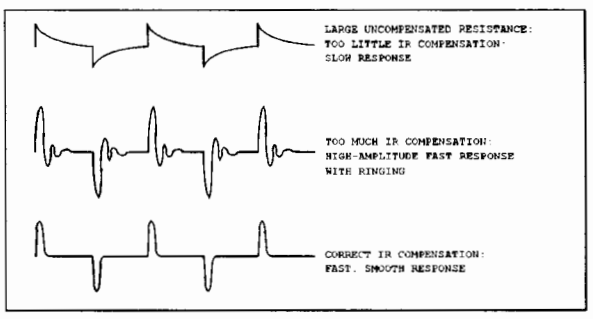


Figure 8-2. I OUTPUT WAVEFORMS AS A FUNCTION OF R_U COMPENSATION (SQUARE WAVE APPLIED)

Figure 8-2 illustrates the effect of the compensation. For optimum stability, the rise time should be as fast as possible without ringing. When the uncompensated resistance is about 90% to 95% compensated, the waveform will show a smooth decay with perhaps just a bit of undershoot. This is optimum for fast measurements.

As the compensation is increased to the 95% to 100% range, increasing ringing appears as the system becomes more unstable. This level of compensation is not suitable for fast measurements. Any data taken during the ring-down will be of doubtful validity. Also, even a slight increase in compensation from this point (or a real decrease in the actual R_U) could cause the potentiostat to oscillate.

Compensation in the 95% to 100% range is best suited to slow experiments (such as for corrosion). Since Current Interrupt Compensation is ideal for slow measurements, there will normally be relatively few instances where Positive Feedback Compensation in the 95% to 100% range would be used.

When you are satisfied with the compensation level, turn off the cell and set up the other parameters you need for the intended measurement. Unless changed, the compensation level you set will remain in memory, ready to be applied whenever Positive Feedback iR Compensation is activated.

Potential, Working Electrode
*See **Cell Potential**.*

Remote Operation
The Model 263A can be controlled from an IBM-compatible personal computer via either its GPIB (IEEE-488) interface or its RS-232 interface. When it is under remote control, its LCD panel displays the REMOTE screen and its front-panel controls, except for the CELL and POWER switches, are inactive.

You can return it to local operation simply by pressing the LOCAL key (F5). But in remote operation via GPIB, pressing F5 is effective only if the LOCAL LOCKOUT message has not been sent from the host computer. This GPIB message would be sent only under special circumstances, and in any case is not supported by EG&G PARC software. See Appendixes A and B of the *Model 263A Command Set Handbook* for a discussion of GPIB communications considerations.

Other ways of returning to local include applying the GO TO LOCAL message, deasserting $\overline{\text{REN}}$, or cycling the power. GO TO LOCAL is another GPIB message defined by IEEE-488 and explained in the *Model 263A Command Set Handbook*.

Reset
In the [System Interface] menu screen, F2 is assigned the "Reset" (Reset) function. This function resets all *experiment* (not communications) parameters to their default values. When you press F2, the message RESET SYSTEM? is displayed. If you want to reset the experiment parameters, press the YES key (F4). All experiment parameter selections will be cleared and default values restored. Note that this will not reset the parameters on the [System Interface] menu (communications parameters and KNOB TIMEOUT).

This effect is not the same as that which occurs when the power is cycled. When the Model 263A powers down, all parameter values and selections are retained. When it is powered up, most of the values in effect at the time of power shutdown are restored.

If you wish to reset *all* parameters, including those on the [System Interface] menu, hold down any front-panel key

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when you power up. Refer to Chapter 5, Section 5.7 for a more detailed explanation of resetting methods.

Reverse Scan
In the Potentiostat Scan and Galvanostat Scan modes, the F5 key is assigned the "Reverse" function. Pressing F5 during a scan reverses the direction of the scan.

Scan Rate
The scan rate is the rate of change of cell potential or current with respect to time. It is set from the [Potentiostatic Scan] or [Galvanostatic Scan] menu screen.

Scan rates from 1 μV/s to 1 V/s can be specified in the [Potentiostatic Scan] mode. In the [Galvanostatic Scan] mode, the range is 1 μX/s to X/s, where X is the selected current range.

Note that the Model 263A does not apply a linear scan, but rather a *staircase scan*. However, as long as the individual steps are very small relative to the range scanned, linear scan theory will apply to a close approximation. When LINE SYNC is not enabled, the nominal step size is the Scan Rate/250, or 250 μV, whichever is larger. LINE SYNC causes larger steps.

Note: Under remote control via the rear panel GPIB or RS-232 port, steps as small as 25 μV (MR = 1), or 2.5 μV (MR = 0), can be attained as explained in the *Model 263A Command Set Handbook*). For example, with a scan rate of 1 V/s, the step size will be 4 mV, because 1000/250 = 4.

Serial Baud Rate
A baud is a unit of signaling speed. In serial RS-232 communications, it refers to the number of times the state or condition of a line changes per second. Baud rates from 110 to 19,200 can be selected from the [System Interface] menu screen. The default setting is 9,600 baud.

Serial Data Bits
Serial communications between the Model 263A and a host computer normally are in ASCII code. You can also select a binary load or dump. From the [System Interface] menu screen, you can select a character length of either seven or eight data bits. Eight data bits are required for binary loads and dumps. If "8" is selected, the Model 263A will always send a "0" as the eighth (MSB) bit when transmitting ASCII codes.

Serial Echo
From the [System Interface] menu screen, you can select either SER. ECHO = OFF or SER. ECHO = ON. If you select ON, each character received via the RS-232 port will be echoed back to the character source. The Serial Echo is normally used only when the Model 263A is connected to a CRT terminal.

Note: Do not confuse this function with the GPIB ECHO.

Serial Parity
In serial RS-232 communications, parity checking is an optional method of error detection. It provides a direct way of detecting garbled data if the computer is programmed to only accept words having the selected parity.

Parity checking traps errors in the serial data stream in the following manner. When the Model 263A frames each character, it counts either the number of 0's or the number of 1's in the data bits. It then appends a parity bit that corresponds to whether the count was even or odd. The host computer also counts the 0's or 1's in the data bits as they arrive, and compares this count with the arriving parity bit. If an error is detected, a flag can be set and retransmission can be requested.

From the [System Interface] menu screen, you can select either SERIAL PARITY = ODD, SERIAL PARITY = EVEN, or SERIAL PARITY = NONE. The parity setting on the Model 263A must match that on the host. If ODD or EVEN parity is selected, the Model 263A will generate the parity bit when transmitting and detect it when receiving.

Serial Stop Bits
This sets the number of stop bits in RS-232 communications. The stop bit or bits signal the end of a binary bit frame, and return the line to the idle state. Stop bits follow the parity bit; either one or two may be used. Two stop bits give more reliable communications at high baud rates.

Step Mode, Potentiostatic or Galvanostatic
The step mode [Potentiostatic Step] or [Gavanostatic Step] substitutes a sharp step between two potential or current levels instead of a gradual scan. The cell potential or current will step as quickly as possible between the two levels. The step transition between levels can take as long as 4 ms, during which no other operation can be started.

The step feature is useful for applying pulse waveforms. For example, it allows the Model 263A to perform double-potential step chronoamperometry, in which the applied potential starts at E INIT, remains there long enough to let the sample come to equilibrium with the solution, steps to E MID for T MID (perhaps 2 seconds or so), and then steps to E FINAL, which continues to be applied.

Stop Bits
*See **Serial Stop Bits**.*

T Init, T Mid, T Final
*See **Delays**.*

Terminator Character
*See **GPIB Terminator Character**.*

Test Echo
*See **GPIB Test Echo**.*

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Update Q With Cell Off

In a perfect instrument and cell system, the accumulated charge Q could not change when the cell is disconnected, because no cell current would flow. But in a real system, no matter how precise, some small charge may appear as a result of drift and other circuit variations. Thus, setting UPDATE Q W/CELL OFF = NO (from the [System Default] menu) prevents charge from accumulating when the cell is off. Choosing YES allows it to accumulate, and might help to estimate drift errors.

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Appendix
Technical Description

A.1 INTRODUCTION

This appendix includes electrical and physical specifications of the standard Model 263A, a somewhat simplified description of its overall architecture and internal circuitry, and a list of the connector pinouts.

A.2 SPECIFICATIONS

Standard Environmental Conditions

Operating Conditions

This equipment shall meet all specified performance criteria when subjected to any natural combination of input voltage, ambient temperature, relative humidty and altitude as defined in the following paragraphs.

Input Voltages

90 to 130 volts rms at 48 to 62 Hz
200 to 260 volts rms at 48 to 62 Hz

Ambient Temperature:

From 10°C to 40°C the instrument shall operate but may not meet some temperature related specifications.

From 20°C to 30°C the instrument shall operate and meet all its specifications over this range.

Relative Humidity: 5% to 85% non-condensing.

Altitude: -500 to 9000 feet relative to sea level.

Non-Operating Conditions

This equipment shall exhibit no significant deterioration of performance after exposure to any natural combination of stress in a non-operating condition.

Ambient Temperature Range: 0°C to 70°C.

Relative Humidity: greater than 95% with modest local condensation or frost formation. Instrument must be allowed to remain at operating conditions for at least 24 hours before applying power.

Altitude: to 50,000 feet.

International Standards

This equipment is designed to meet or exceed the requirements of the following standards:

BS ENN55022 (1987), Class B
BS EN50082 (1992):
IEC 801-2:1991
IEC 801-3:1994
IEC 801-4:1988
BS EN61010-1 (1995), Installation Category II, Pollution Degree 2.

Performance Specifications

Power Amplifier

Compliance Voltage: > ±20 V
Maximum Output Current: > ±200 mA
(> ±2 A with 94 Option)
Slew Rate: 1 V/μs
Bandwidth (Open Loop Unity Gain): >200 kHz
Voltage Temperature Stability: <50 μV/deg C

Differential Electrometer

Input Impedance: $>10^{10}\Omega$ in parallel with 20 pF
Input Bias Current: <50 pA at 25 deg C
Maximum Input Voltage
Differential: ±10 V
Reference Input: ±11 V
Common Mode Rejection
>70 dB at 100 Hz
>60 dB at 100 kHz
Bandwidth: -3 dB at >4 MHz
Offset Voltage: <100 μV
Offset Temperature Stability: <50 μV/deg C

iR Compensation

Positive Feedback
Range: 20 MΩ to 20 Ω depending on current range
with 91 Option: 20 MΩ to 2 Ω
Resolution: 0.05% of current range
Current Interrupt
Digital Potential Error Correction: 12 bit DAC
Total Interruption Time: <50 μs to 2050 μs

Current Measurement

Ranges: 7 decades, 100 mA to 100 nA
with 94 option: 8 ranges, 1 A to 100 nA
Accuracy (dc) at I OUTPUT BNC
10 μA to 100 mA Ranges: <0.4% Full Scale
with 94 option: highest range is 1 A and accuracy is <0.4% Full Scale
100 nA and 1 μA Ranges: <0.5% Full Scale, ±5 nA
Frequency Response (Small Signal)
1 mA Range: -3 dB at 100 kHz, 1 kΩ source impedance
10 μA Range: -3 dB at >4 kHz, 100 kΩ source impedance

Potential/Current Control

Bias Digital/Analog Converter (DAC)
Resolution: 14 bits
Range (Potentiostat): ±8 V

Modulation Digital/Analog Converter (DAC)
Resolution: 14 bits

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Range (Potentiostat): ±2 V, ±0.2 V, and ±0.02 V
Range (Galvanostat): ±200%, ±20.00%, and ±2.000% of Full-Scale current

With 91 Option:
Resolution: 16 bits
Range (Potentiostat): ±8 V, ±0.8 V, and ±0.08 V
Range (Galvanostat): ±200%, ±20.0%, and ±2.00% of full-scale current

Accuracy
Applied Potential: 0.2% of reading ±2 mV
Applied Current: 0.2% of Full-Scale current

System
Rise Time (10% to 90%)
No Load: <1 μs
10 kΩ, 100 μA: <1.25 μs
10 Ω, 100 mA: <5 μs
Noise and Ripple: typically <50 μV rms referred to External Input
Bandwidth (E_{in}/E_{out}), 1 mA Full-Scale current range, 1 kΩ resistive load: -3 dB at >10 kHz

Computer Interfaces
RS-232C
IEEE 488.1-1987 with the following interface functions implemented: SH1, AH1, T6, L4, SR1, RL1, PP0, DC1, DT0, C0, E2

Weight
10 kg (22 lb)

Size
17.5 in. W × 5.5 in. H × 18.5 in. D

Power Requirements
90–130 VAC or 200–260 VAC, 50–60 Hz, 125 watts maximum

A.3 CIRCUIT DESCRIPTION

In essence, the Model 263A I/E Converter and its interfacing circuitry is a high-current operational amplifier. Its basic function depends on the operating mode. In potentiostatic operation (Figure A-1), the Model 263A converts the cell current to a proportional voltage. In galvanostatic operation (Figure A-2), the control loop is configured to cause a cell current equal to the Model 263A's output current.

The microprocessor incorporated in the instrument allows cell voltage and/or current readings to be transmitted to a host computer via a GPIB or RS-232 interface, and allows for control of the system either from the front panel or from a host computer.

In either potentiostatic or galvanostatic mode, the output of two digital-to-analog converters, the Bias DAC and the Modulation DAC, can be summed with a control potential applied at the EXT INPUT conector to control the cell potential or current. The Bias DAC sets the background level. The Modulation DAC allows complex time-varying control programs to be applied. The program control and data-taking processes are coordinated so that the resulting data sets can be plotted or otherwise processed as a function of the applied control program.

The Multiplexer determines whether a voltage or current sample will be applied to the A/D (analog to digital) Converter. The output of this circuit is a signed number proportional to the current or voltage sample applied to its input. This number is read by the microprocessor and sent to the front-panel display at the proper time. In remote operation, it is also stored for transmission to the host computer.

Figure A-1 illustrates potentiostatic operation. As shown, the net control potential (External Input, when applied, and DACs) is summed with the Working Electrode potential detected by the Reference Electrode and buffered by the Electrometer. As a result of the feedback, the output of the summing Power Amplifier drives the Counter Electrode to whatever potential is required to maintain 0 V at the inverting input of the Power Amplifier. This condition is satisfied when the detected Working Electrode potential equals the net control potential. Thus the Working Electrode potential is controlled at the set or programmed level.

Provision is made for monitoring the resulting current. A resistance determined by the selected current range is connected between the output of the I/E Converter amplifier and its inverting input. The Working Electrode current is applied to the inverting input of this amplifier as well, causing it to act as a summing junction. The amplifier output assumes whatever potential it must to force the inverting input potential to that of the noninverting input (ground). This requirement is satisfied when the output voltage is I × R volts, where I is the working electrode current in amperes and R is the feedback resistance in ohms determined by the current range. Thus the current range determines the amount of cell current that will give a full-scale (1 V) output. A 1 V output corresponds to a Working Electrode current of 100 nA, 1 μA, 10 μA, 100 μA, 1 mA, 10 mA, or 100 mA, according to the selected current range.

The signal conditioning circuitry denoted by a block in Figures A-1 and A-2 is detailed in Figure A-3.

In galvanostatic operation (Figure A-2), the Model 263A high-current I/E Amplifier is ahead of the summing Power Amplifier, instead of after it as in potentiostatic operation. The I/E Amplifier is connected as a gain-of-one inverting amplifier. A potential equal to the net control potential but of opposite polarity appears at its output. The potential is applied to the resistance determined by the selected Current Range, and, because the other end of the selected range resistor is at ground, a current of E/R amperes occurs, where R is the selected range resistor and E is the net control potential.

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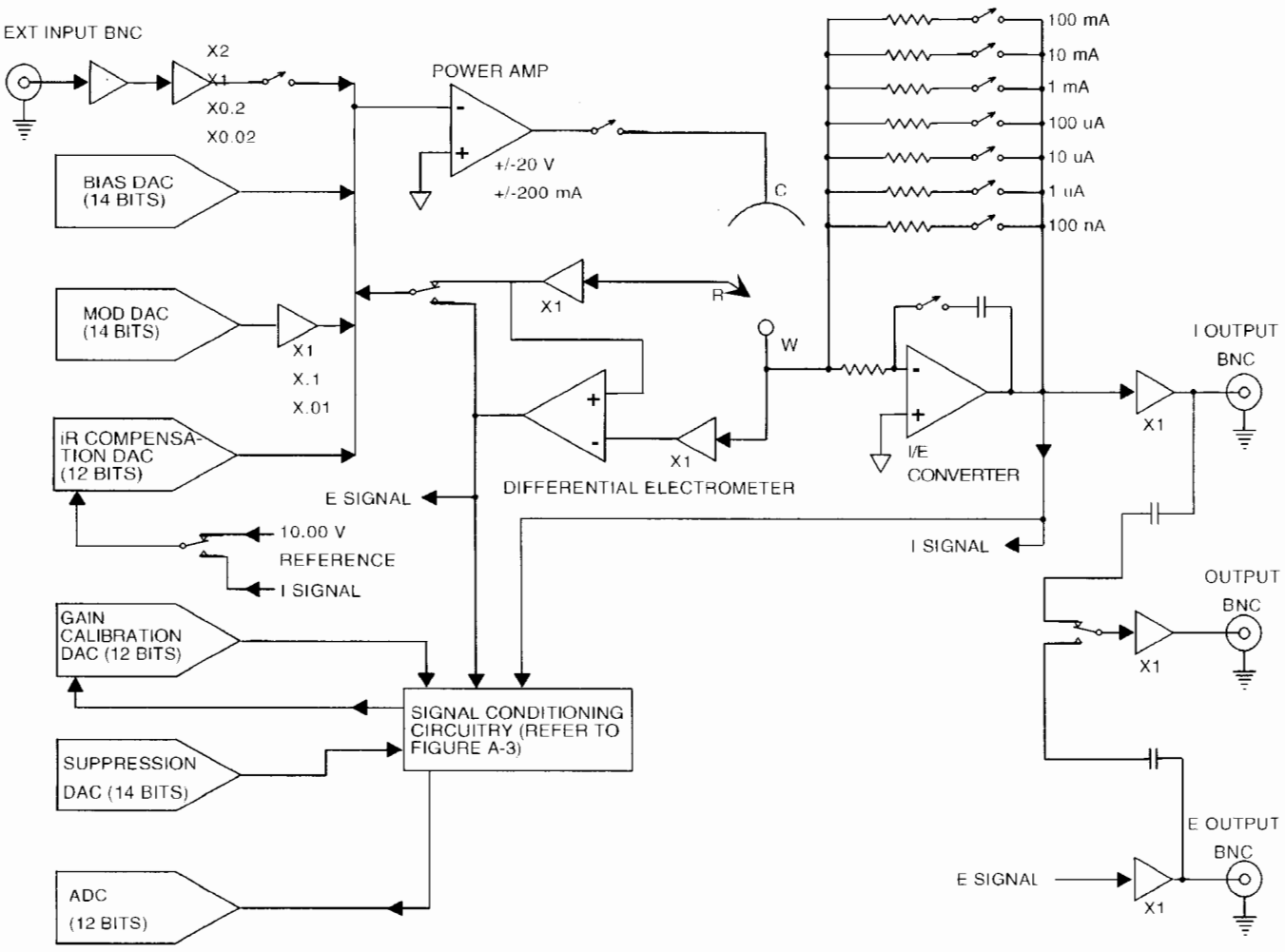


Figure A-1. SIMPLIFIED BLOCK DIAGRAM OF MODEL 263A IN POTENTIOSTATIC MODE

The output of the summing Power Amplifier drives the cell Counter Electrode. All of the current through the Current Range resistor goes through the cell. Thus the current to which the cell is controlled is determined by the current range selected as well as by the control potential (External Input, when applied, and DACs).

As shown in Figure A-2, the potential can be monitored by the Reference Electrode and reported, if desired. Although this feature adds flexibility and provides important information in certain applications, it is not essential to galvanostatic operation.

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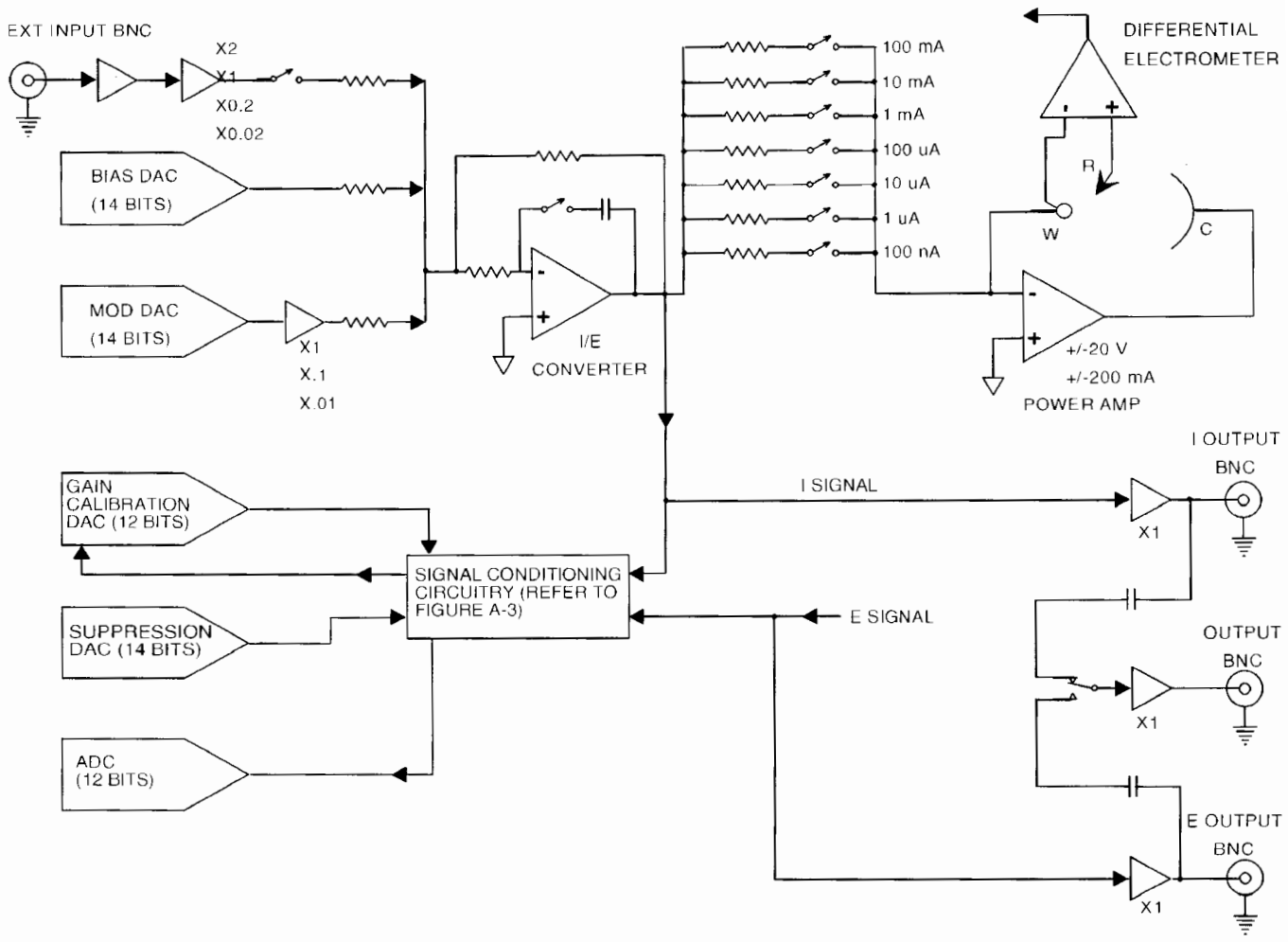


Figure A-2. SIMPLIFIED BLOCK DIAGRAM OF MODEL 263A IN GALVANOSTATIC MODE

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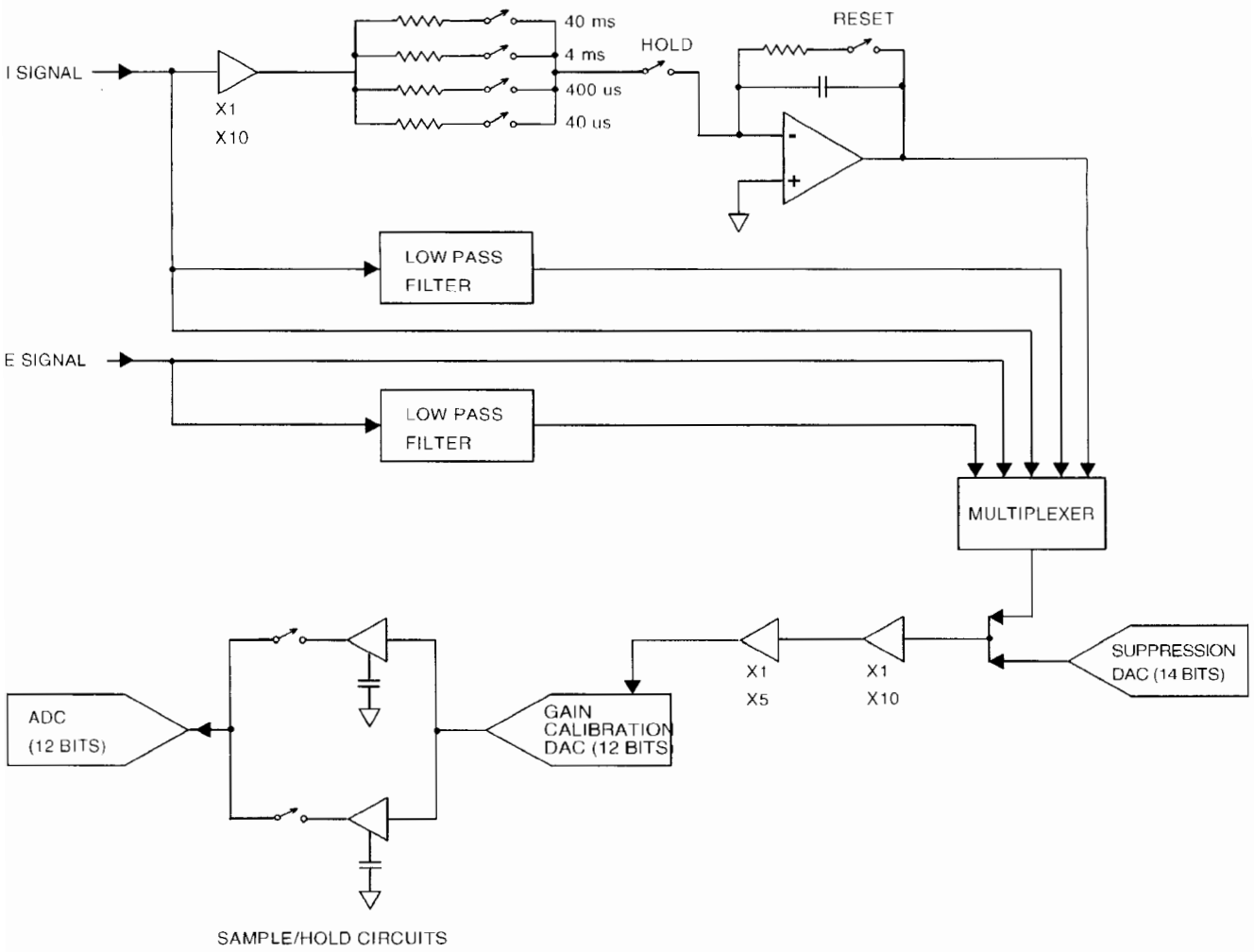


Figure A-3. SIMPLIFIED BLOCK DIAGRAM OF SIGNAL CONDITIONING CIRCUITRY

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A.4 263A/98 OPTION

The 98 Option, depicted in Figure A-4, provides three additional functions to the Model 263A that significantly extend its capabilities. First, this option allows suppression of any I and E dc signal present at the Model 263A front-panel input. Second, the 98 Option allows an auxiliary signal applied to the rear-panel AUX IN 1 or AUX IN 2 BNC connectors to be monitored. As shown in Figure A-4, a multiplexer is provided so that either auxiliary input can be monitored. Third, an OUTPUT DAC is provided that allows a precise dc voltage in the range of ±2 V to be generated internally and made available at the rear-panel OUTPUT 1 connector. *OUTPUT 1 only is supported at this time.*

The rear-panel AUX IN 1, AUX IN 2, OUTPUT 1 and OUTPUT 2 connectors, although present in the base unit, are not connected or active unless the 98 Option has been installed. The 98 Option's suppression function acts through the front-panel E OUTPUT and I OUTPUT connectors, which are also present and active in the base unit, but without the suppression capabilitiy.

The 98 Option is controlled from an external computer using the command set described in the *Model 263A Command Set Handbook.*

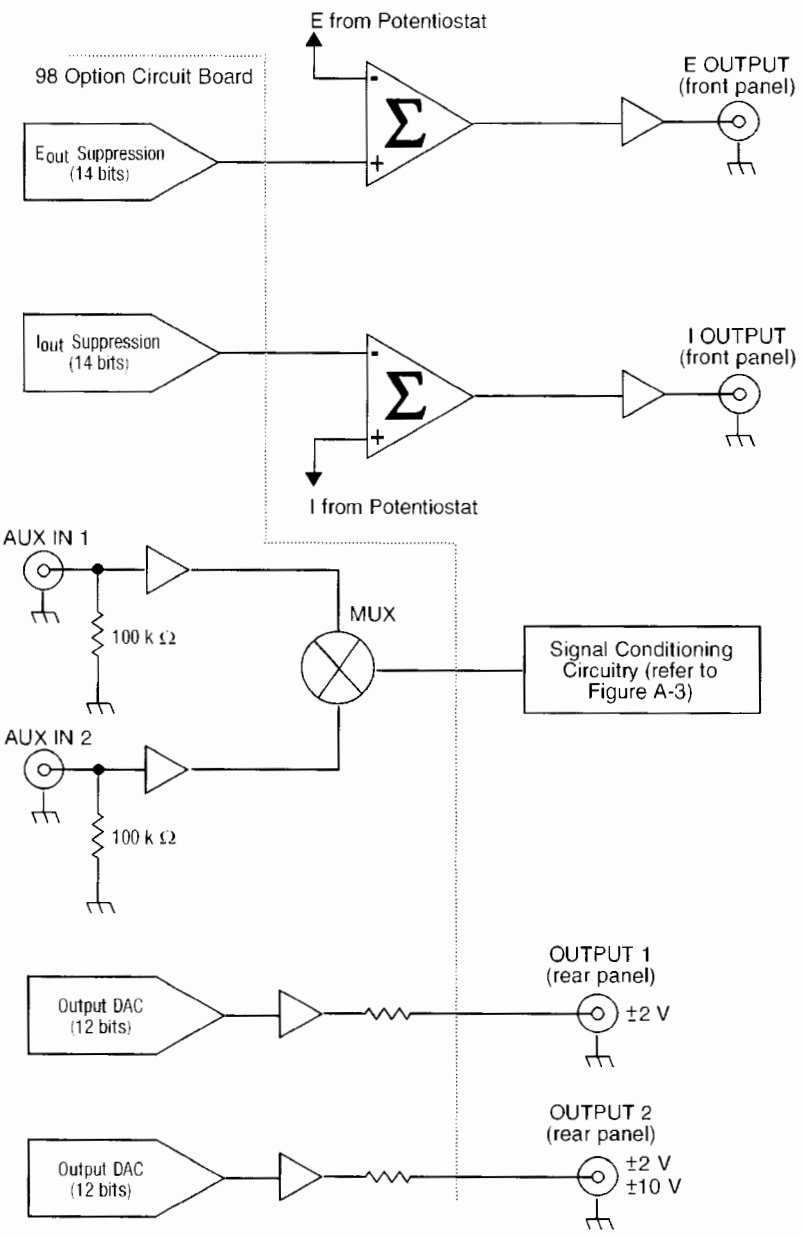


Figure A-4. MODEL 263A/98 OPTION BLOCK DIAGRAM

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A.5 263A/99 OPTION

The 99 Option, depicted in Figure A-5, incorporates all of the features of the 98 Option, described in the previous paragraph, plus optical couplers that provide isolation, allowing floating operation.

Physically, the 99 Option consists of two circuit boards. One of these, the Opto-Coupler board, contains the opto-couplers that provide isolation of the I/O Devices (RS232 and GPIB communications) and Processor. The other board is an Isolation board. It has the same functions as the 98 Option but also provides isolation of measurement signals on the front and rear of the 263A. ***Note:*** *The isolation board is not freely interchangeable with the board provided with the 98 Option.*

In FLOAT operation (rear-panel Float/Normal switch set to **FLOAT**), the measurement ground is at the potential of the black cell-cable lead. When the Float/Normal switch is set to **NORMAL**, the black lead is at earth ground (potential of the ground pin of the power-cord plug), establishing conventional operation but with all of the 98 Option features enabled.

For example, in doing a corrosion study of a tank buried in the ground, by operating in the **Float** mode, the black lead could be attached directly to the tank and the measurement potentials would be with respect to the tank reference. There might be situations where the black lead would be connected to a special electrode to reference the 263A to a convenient, if not precisely defined, local ground. For example, if you were making measurements on a group of objects in a solution and simply connected the black lead to a carbon electrode in the solution near the objects, the 263A measurement circuitry would assume the potential at the carbon rod as its "ground".



WARNING! A unit operated in Float mode (set via the rear-panel FLOAT-NORMAL switch) may have potentials of up to ±300 V on any or all pins of the Accessory Power and Cell connectors, depending on the nature and configuration of the cell to which the unit is connected. Extreme caution should be exercised when inserting or removing these connectors in a floating system.

A system that is floating can be very dangerous to work with. If the system is floated at a dangerous potential (possible but not typical in electrochemical and corrosion measurements), accidental contact with a point at the elevated potential could result in dangerous, even lethal, electrical shock.

A.6 263A/94 OPTION

This option consists of modifying the 263A's Power Amplifier so as to increase it's current output capabilities from ±200 mA to ±2 A. Besides adding the higher-current output stages, this option includes installation of a fan that vents through an opening at the rear panel. The fan used is a variable-speed type that automatically speeds up on sensing the higher temperatures characteristic to high-power operation. Note that the only air intakes for the fan are on the right side of the 263A as viewed from the front. It is absolutely essential that the air flow path to these vents be kept clear. This is particularly critical when the 263A is mounted in a cabinet or rack.

A.7 263A/91 OPTION

The 91 Option provides faster acquisition times (30 μs vs 100 μs for the standard unit), 96 k of additional RAM in the data buffer, and 16-bit DACs, enabling ±8 V scans from the initial potential (within the 263A's ±10 V hardware limit).

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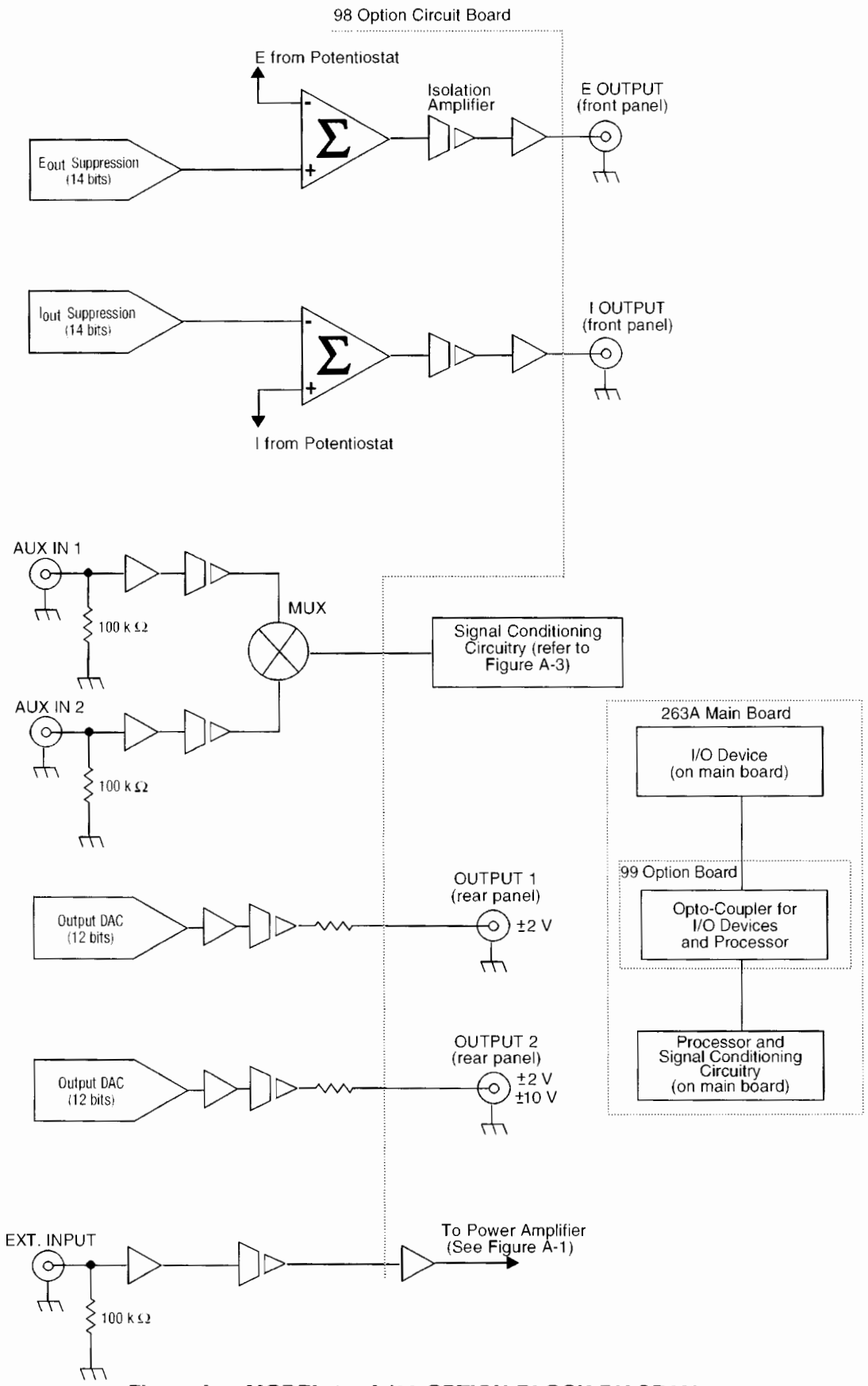


Figure A-5. MODEL 263A/99 OPTION BLOCK DIAGRAM

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A.8 CONNECTOR PINOUTS

Cell Connector (7-Pin LEMO Connector)

PIN	FUNCTION
1	Counter Electrode
2	Analog Ground
3, 4	Working Electrode
5	Working Electrode Shield
6	Reference Electrode
7	Reference Electrode Shield



Potentials as high as ±20 V at currents as high as 200 mA (2 A with 94 option) may be present at the counter electrode lead of the cell cable. **The high current capability requires that reasonable care be taken in handling these leads**. The CELL switch should always be off when making the cell connections (its indicator should not be lit). It should similarly be off when the cable leads are being examined or disconnected.

Voltage Levels on Systems Equipped with /99 Option: A unit operated in Float mode (set via the rear-panel FLOAT-NORMAL switch) may have potentials of up to ±300 V on any or all pins of the Cell connector, depending on the nature and configuration of the cell to which the unit is connected. Extreme caution should be exercised when inserting or removing these connectors in a floating system.

Auxiliary Interface (Mating Connector DB9S)
This connector provides several functions only available when the Model 263A is being controlled from an external computer via the RS-232 or GPIB (IEEE-488) port. Included are the signals required to drive a Model 303A Static Mercury Drop Electrode. If this electrode is used, connections between the Auxiliary Interface and the Model 303A are made via the Model 507 Interface.

The Model 507 Interface is a universal power supply and control interface for the Model 303A SMDE. It can relay DISPENSE, PURGE, and STIR signals from the Model 263A AUXILIARY INTERFACE connector to the Model 303A. The Model 507 is provided with all cables required to make the necessary connections. Refer to the *Model 507 Interface Installation Guide*, EG&G PARC Part Number 222556, for installation instructions.

A pinout list for the AUXILIARY INTERFACE connector follows.

PIN	SIGNAL	FUNCTION
1	ground	
2	EXT TRIG	This input allows an operation in progress to be halted via the WFT command from an external computer. If the command WFT 0 is applied, operation will resume when a TTL logic 0 is applied to the EXT TRIG line. If the command WFT 1 is applied, operation will resume when a TTL logic 1 is applied to the EXT TRIG line.
3	$\overline{\text{DISPENSE}}$	In applications where the Model 263A is being used with a Model 303A via a Model 507 Interface, this signal causes the Model 303A to do a Dislodge/Dispense operation on command from an external computer. The computer initiates the dispense operation by applying a DISP command (see description in the *Model 263A Command Set Handbook*).
4	TRIG OUT	A TTL trigger output is provided on this line on application of the TRIG command. The Trig Out baseline is either a logic 0 or a logic 1 (default is logic 0), according to the operand of the last applied TRIG command. TRIG 0 establishes a logic 1 baseline. TRIG 1 establishes a logic 0 baseline. Once a logic 0 baseline is established, subsequent TRIG 1's will cause a 10 to 20 ms wide logic 1 pulse to be generated. Similarly, once a logic 1 baseline is established, TRIG 0's will cause a 10 to 20 ms wide logic 0 pulse to be generated.
5	BIT 0 IN	The level on this line will be read and reported to the host computer if the command BIT 0 is applied.
6	+5 V	+5 V at up to 100 mA is available for external use.
7	BIT 0 OUT	If Current Interupt iR Compensation is *not* active, the level on this line will be set to a logic 0 if the command BIT 0 0 is applied. It will be set to a logic 1 if the command BIT 0 1 is applied.
		If Current Interrupt iR Compensation is active, this line will go high about 3 μs before the start of the current interruption and will go low about 7 μs after the end of the current interruption. This pulse can be used to trigger an oscilloscope so that the iR Current Interrupt wave-

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form can be easily monitored at the E OUTPUT connector.

8 $\overline{\text{PURGE}}$ — When operating a Model 263A with a Model 303A SMDE via a Model 507 Interface, this signal controls the Model 303A's purge function in response to commands from the external computer. The computer initiates a purge by applying a PURGE command (see discussion of this command in the *Model 263A Command Set Handbook*).

9 STIR — This signal controls the Model 303A's Stir function in response to commands from an external computer. The assumption is that the Model 303A is being used with a Model 305 Stirrer.

Accessory Power Connector (5-Pin DIN Connector)
Note that the pins on this DIN connector are not numbered consecutively. They are numbered in the following sequence, looking into the connector from the rear of the instrument and counting clockwise from the keyway at the top.

PIN	FUNCTION
1	-26 VDC Regulated
4	-15 VDC Regulated
2	Common
5	+15 VDC Regulated
3	+26 VDC Regulated



Voltage Levels on Systems Equipped with /99 Option:
A unit operated in Float mode (set via the rear-panel FLOAT-NORMAL switch) may have potentials of up to ±300 V on any or all pins of the Accessory Power connector, depending on the nature and configuration of the cell to which the unit is connected. Extreme caution should be exercised when inserting or removing these connectors in a floating system.

IEEE-488 GPIB Interface

PIN	FUNCTION
1	$\overline{\text{DIO1}}$
2	$\overline{\text{DIO2}}$
3	$\overline{\text{DIO3}}$
4	$\overline{\text{DIO4}}$
5	$\overline{\text{EOI}}$
6	$\overline{\text{DAV}}$
7	$\overline{\text{NRFD}}$
8	$\overline{\text{NDAC}}$
9	$\overline{\text{IFC}}$
10	$\overline{\text{SRQ}}$
11	$\overline{\text{ATN}}$
12	SHIELD
13	$\overline{\text{DIO5}}$
14	$\overline{\text{DIO6}}$
15	$\overline{\text{DIO7}}$
16	$\overline{\text{DIO8}}$
17	$\overline{\text{REN}}$
18	GND 6
19	GND 7
20	GND 8
21	GND 9
22	GND 10
23	GND 11
24	LOGIC GROUND

RS-232C Interface

PIN	FUNCTION	COMMENT
1	Chassis Ground	Ties the chassis of the Model 263A to that of the computer.
2	Transmit Data	The Model 263A transmits on this line. It must connect to the computer connector pin that receives serial data.
3	Receive Data	The Model 263A receives data on this line. It must connect to the computer connector pin that transmits serial data.
4	Request to Send	This line is permanently asserted in the Model 263A, that is, it is (always at +12 V). The Model 263A is ready to receive a character.
5	Clear to Send	Computer controled line. To enables 263A to transmit, the line is placed at the positive logic level (+3 V to +12 V). To hold off transmission by the Model 263A, the line must be at the negative logic level (-3 V to -12 V). If left unconnected, the Model 263A is allowed unimpeded transmission.
6	Unused	
7	Logic Ground	Data signal levels should be with reference to logic ground. The logic ground line of the Model 263A should interconnect with the logic ground line of the computer.
8-25	Unused	

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Note that the Model 263A RS-232 port is configured as a female DTE port (Data Terminal Equipment) rather than as a DCE (Data Communications Equipment). Thus, cabling to most microcomputers can be accomplished with a standard reversed or switched RS-232 cable, also known as a null modem cable.

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