# Model 273A Potentiostat/Galvanostat

**User's Manual** 

# **Advanced Measurement Technology, Inc.**

a/k/a Princeton Applied Research, a subsidiary of AMETEK®, Inc.

# WARRANTY

Princeton Applied Research\* warrants each instrument of its own manufacture to be free of defects in material and workmanship. Obligations under this Warranty shall be limited to replacing, repairing or giving credit for the purchase price, at our option, of any instrument returned, shipment prepaid, to our Service Department for that purpose within ONE year of delivery to the original purchaser, provided prior authorization for such return has been given by an authorized representative of Princeton Applied Research.

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- A. Contact the Customer Service Department (865-482-4411) or your local representative to discuss the problem. In many cases it will be possible to expedite servicing by localizing the problem.
- B. If it is necessary to send any equipment back for service, we need the following information.
  - 1. Model number and serial number.

2. Your name (instrument user).

- Your address.
- 4. Address to which the instrument should be returned.
- 5. Your telephone number and extension.
- 6. Symptoms (in detail, including control settings).
- 7. Your purchase order number for repair charges (does not apply to repairs in warranty).
- 8. Shipping instructions (if you wish to authorize shipment by any method other than normal surface transportation).
- C. U.S. CUSTOMERS Ship the equipment being returned to:

Advanced Measurement Technology, Inc. PHONE: 865-482-4411 801 S. Illinois Avenue FAX: 865-483-2133

Oak Ridge, TN 37831 ATTN: Customer Service

D. CUSTOMERS OUTSIDE OF U.S.A. — To avoid delay in customs clearance of equipment being returned, please contact the factory or the nearest factory distributor for complete shipping information.

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# **Safety Instructions and Symbols**

This manual contains up to three levels of safety instructions that must be observed in order to avoid personal injury and/or damage to equipment or other property. These are:

**DANGER** Indicates a hazard that could result in death or serious bodily harm if the safety instruction is not

observed.

**WARNING** Indicates a hazard that could result in bodily harm if the safety instruction is not observed.

**CAUTION** Indicates a hazard that could result in property damage if the safety instruction is not observed.

Please read all safety instructions carefully and make sure you understand them fully before attempting to use this product.

# **Cleaning Instructions**

**WARNING** Using this instrument in a manner not specified by the manufacturer may impair the protection provided by the instrument.

#### To clean the instrument exterior:

- Unplug the instrument from all voltage sources.
- Remove loose dust on the outside of the instrument with a lint-free cloth.
- Remove remaining dirt with a lint-free cloth dampened in a general-purpose detergent and water solution. Do not use abrasive cleaners.

**CAUTION** To prevent moisture inside of the instrument during external cleaning, use only enough liquid to dampen the cloth or applicator.

• Allow the instrument to dry before reconnecting the power cord.



# 1. INTRODUCTION

#### 1.1. Overview

The Model 273A Potentiostat/Galvanostat features both front-panel and computer control for virtually unlimited flexibility and utility. It uses the latest analog and microcomputer design advances to provide high performance, ease of use, and greater versatility in electrochemical measurements.

The instruments 100 V compliance and 1 A output capability allows rapid and accurate potential or current control in virtually any electrochemical cell. The high compliance voltage is particularly important in maintaining potential control when working with high-resistance, dilute electrolytes or non-aqueous solvents. Moreover, it enables faster charging of the cell capacitance, allowing the potentiostat to rapidly respond to step transitions in the control potential.

Component selection, shielding, grounding, and circuit design are all carefully optimized for minimum internal electronic noise, giving high sensitivity and a quiet output signal. A low pass filter facilitates dealing with noise arising in the cell itself.

The Model 273A uniquely addresses the critical question of stability versus speed. The user can select a HIGH SPEED mode to take advantage of the potentiostat's extraordinarily fast rise time. Alternatively, the user can select the HIGH STABILITY mode for oscillation-free operation with almost any cell.

A high-performance, four-terminal current-to-voltage converter circuit is inducted for accurate, rapid, low-drift current measurements, free of degradation from cell-cable resistance, capacitance, and inductance.

The Model 273A uses an external differential electrometer. Not only does this approach further assure freedom from cell-cable effects, but it allows the use of two reference electrodes to control the potential across an interface, such as a membrane. In addition, the electrometer can be configured to provide a remote sensing contact to the working electrode and so eliminate any potential error resulting from high current in the contact resistance.

#### 1.2. About this Manual

The Model 273A Potentiostat/Galvanostat can be operated either directly with its front-panel controls or remotely from a personal computer or workstation. This *Instruction Manual* provides details of the physical and electrical characteristics of the Model 273A, and describes how to operate it as a stand-alone instrument controlled from its front panel.

Instructions for operating the unit remotely via either the RS-232C or GPIB (IEEE-488) interface port are given in the separately bound Model 273A Remote-Programming Command Handbook. In addition to the command descriptions, the Command Handbook gives detailed explanations of GPIB (IEEE-488) and RS-232C communications, including rear-panel switch settings, communications protocols, and some useful communications routines. Also included in the Command Handbook is an application note on waveform programming, one of the most useful functions of the Model 273A.

This Instruction Manual is organized into five chapters and three appendices. Chapter 1 provides a general description of the Model 273A. Be sure you understand the information in Section 1.4 about the polarity convention used in this instrument.

The 100 V output and high current capability of the Model 273A make it potentially lethal if not used with care and respect. Chapter 2 describes recommended safety precautions for operating it. Chapter 2 also explains how to set the instrument for operation with different input power voltages, replace the power line fuse, and determine whether power is applied to the counter electrode connector.

Chapter 3 describes the physical and electrical characteristics of the instrument. It includes a description of the electrical circuitry and internal organization of the instrument, and the functions of the front- and rear-panel connectors. Pinouts of the connectors are given in Appendix A, and schematic diagrams and component layouts are shown in Appendix C. After you inspect the Model 273A for shipping damage, but before you begin to use it, run the initial performance checks given in Chapter 4 to ensure that the instrument operates correctly. Then, using the operating instructions given in Chapter 5, you may begin operation of your unit.

The Model 273A may be mounted in a standard 19-in. (475 cm) rack assembly. Appendix B provides mounting instructions.

## 1.3. Controlling the Model 273A

#### **Front-Panel Controls**

The front panel is configured for maximum versatility in experiment definition. An optimally human-engineered Scan Setup section is provided to allow cyclic staircase. voltammetry, corrosion experiments, and other pulse and staircase waveforms to be easily performed. The alphanumeric liquid crystal display assists in setting up an experiment and in presenting real-time data. A Scan Status Display additionally provides continuously updated status information as the experiment advances. A simple touch of a button implements such advanced features as automatic current ranging and current-interrupt IR compensation. BNC connectors on the front and rear panels make all pertinent signals available for analog recording.

#### **Remote Computer Control**

By interfacing the Model 273A to an external computer via the GPIB or RS-232C Interface, complete remote control of the instrument is readily accomplished using the Electrochemical Command Set, a group of over 100 mnemonic software instructions specifically developed for electrochemical measurements. These commands place unprecedented flexibility in the hands of the electrochemist. They provide:

- Access to all 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.

These commands and all necessary instructions for operating the Model 273A via an external computer are described in the separately bound *Model 273A Remote-Programming Command Handbook*.

The Model 273A is fully equipped with the necessary hardware to implement experiments from your computer, including:

- Two 14-bit digital-to-analog converters for versatile waveform generation.
- A 12-bit analog-to-digital converter to measure current and potential.
- 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.

## 1.4. Polarity Convention

The Model 273A follows the American polarity convention and the display indications are consistent with that convention. Positive current is catholic, that is, a current is defined as positive if reduction is taking place. Negative current is anodic, that is, a current is defined as negative if oxidation is taking place.

In potentiostatic operation, making the applied potential more positive will make the current tend to be more anodic. Conversely, making the applied potential more negative will make the current tend to be more catholic. This is true for all potential sources, including EXTERNAL INPUT.

In galvanostatic operation, making the applied current more positive by any means except applying a potential to the EXTERNAL INPUT will tend to make the current more catholic. Making the applied current more negative will tend to make the current more anodic. This sense is reversed at the front panel external input. Them 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 the EXTERNAL INPUT is a high-impedance (100 k $\Omega$ ) input in both potentiostatic and galvanostatic operation. (In galvanostatic operation, 1 V applied results in a full-scale current.)

# 1.5. Inspection of New Instrument

Newly received apparatus should be inspected for shipping damage. If any is noted, immediately notify Princeton Applied Research and file a claim with the carrier. Save the shipping container for possible inspection by the carrier.

Warning! The protective grounding could be rendered ineffective in damaged apparatus.

Damaged apparatus should not be operated until its safety has been verified by qualified service personnel. Damaged apparatus should be tagged to indicated to a potential user that it may be unsafe and that it should not be operated.

#### 1.6. Maintenance and Service

The Model 273A Potentiostat/Galvanostat has been designed for optimum reliability, and requires no periodic maintenance.

This manual contains no service information. The Model 273A 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.

There are no operator serviceable parts inside. Refer servicing to qualified personnel.

# 2. SAFETY CONSIDERATIONS

#### 2.1. Introduction

The apparatus to which this instruction manual applies has been supplied in a safe condition. This manual contains some information and warnings that have to be followed by the user to ensure safe operation and to retain the apparatus in a safe condition. The described apparatus has been designed for indoor use.

# 2.2. Safety Mechanism

As defined in EEC Publication 348, *Safety Requirements for Electronic Measuring Apparatus*, the Model 273A is Class I apparatus, that is, apparatus that depends on connection to a protective conductor to earth ground for equipment and operator safety. Before any other connection is made to the apparatus, the protective earth terminal must be connected to a protective conductor. The protective connection is made via the earth ground prong of the Model 273A's power cord plug. This plug shall only be inserted into a socket outlet provided with the required earth ground contact. The protective action must not be negated 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.

The power cord plug provided is of the type illustrated in Fig. 1. If this plug is not compatible with the available power sockets, the plug or the power cord should be replaced with an approved type of compatible design.

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.

In many parts of the world, commonly used power plugs and sockets differ from those in general use in the United States. For this reason, cords supplied with units to be used outside the United States may not be furnished with a power plug, making it necessary for the user to obtain and install a power cord plug suited to use in their area.

For safety, it is necessary that normal polarity relationships be maintained. The wires in the supplied power cord are color-coded for this purpose. 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. Power Voltage Selection and Line Fuses

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

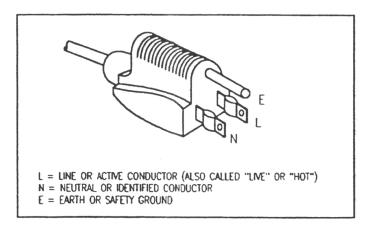


Fig. 1. Power Cord Plug with Polarity Indication.

**Caution!** The apparatus described in this manual may be damaged if it is set for operation from 110 V ac and turned on with 220 V ac applied to the power input connector.

A detailed discussion of how to check and, if necessary; change the power-voltage setting follows.

The Model 273A can operate from any of four different power-voltage ranges, 90-110 V, 110-130 V, 210-230 V, and 230-260 V, 48-62 Hz. Change from one voltage range to another is made by repositioning a plug-in circuit card in the rear panel Line Cord/Fuse Assembly. Instruments are ordinarily shipped ready for operation from 110-130 V ac, unless destined for an area known to use a line voltage in a different range. If this is the case, they are shipped configured for operation from the other range.

If necessary, the change from one range to another can be accomplished in the field. Changing the voltage range or changing the line fuse should only be done by a qualified service technician, and then only with the instrument disconnected from all sources of power. Observing the instrument from the rear, note the clear-plastic "door" immediately adjacent to the power cord connector (Fig. 2). When the power cord is disconnected from the rear-panel connector, the plastic door is free to slide to the left, giving access to the fuse and to the voltage selector circuit card.

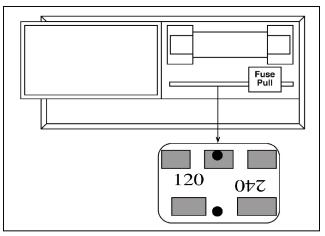


Fig. 2. Power Input Assembly.

The selector card is located at the lower edge of the fuse compartment. A number printed on the upper surface of the selector card is visible without removing the card. The number is somewhat obstructed by the fuse but can be read if the viewing angle is just right. This number indicates the selected nominal line voltage. There are four numbers on the card, but only one is visible. In other words, the card can be inserted in any of four different positions, and a different number can be read in each. A table printed on the rear panel of the Model 273A adjacent to the power-input assembly indicates the actual line voltage range for each number.

If the number showing is incorrect for the prevailing line voltage, the card will have to be repositioned, as follows. The first step is to remove the fuse. When the lever labeled FUSE PULL is rotated out and towards the left, the fuse will lift so that it can be easily removed. At the front center of the circuit card is a small hole that serves as a convenient pry point. Needle-nose pliers or a paper clip can be used as an aid in removing the board. With the board removed, four numbers become visible: 100, 120, 220, and 240. Orient the board so that the desired number (Table 1) will be visible when the board is inserted. Then insert the board into its connector. The selected number should be the only one that shows. Be sure the board is securely seated in its connector.

# Table 1. Voltage-Selection Card Position as a Function of Line Voltage

SELECTOR CARD	OPERATING	
NUMBER EXPOSED	<b>VOLTAGE RANGE</b>	
100	90-110V	
120	110-L30 V	
220	210-230 V	
240	230-260 V	

Next, check the fuse rating. For operation from a line voltage in either of the two lower ranges, use a slow-blow fuse rated at 4A (voltage rating 125 V or higher). For operation from a line voltage in either of the two higher ranges, use a slow-blow fuse rated at 2 A (voltage rating of 250 V or higher). When the proper fuse has been installed, slide the plastic door back over the fuse compartment so that the power cord can be reconnected.

Make sure that only fuses with the required current rating and of the speed type are used for replacement. The use of makeshift fuses and the short-circuiting of fuse holders are dangerous.

#### 2.4. Cell Cable

# **Warning!** Potentials as high as 100 V capable of delivering currents as high as 1 A may be present at the cell connections. To avoid the possibility of dangerous electrical shock, always have the cell disabled when working with the cell connections. The cell status is determined by the two keys in *the cell* area of the Model 273A front panel. These keys function as follows.

There are two keys and one indicator in this group. All three are associated with the internal relay that provides the connection to the counter electrode at the cell. The CELL ENABLE key has two positions, in (ON) and out (OFF). CELL ENABLE is a hard-wired fail-safe switch. Whenever it is in the OFF position, there is no possible way, including commands from an external computer, for voltage to be applied to the counter electrode. This switch is provided to assure operator safety when working on the cell.

Warning!	Anytime work at the cell is done, including making and breaking connections, this
	switch absolutely should be in the off position. The 100 V output and high current
	capability of the 273A make it a dangerous device, one capable of causing severe
	injury or even death.

The upper Cell key provides the normal Cell ON/OFF control during operation. Unlike the CELL ENABLE switch, this key is subject to software override. For example, the ON state must be selected before starting a scan. However, a scan may contain delay intervals to be carried out in the Cell OFF state. If this is the case, the cell is turned off automatically at the appropriate points in the scan sequence.

The cell status can be noted at any time by observing the Cell indicator. This indicator is unique, first in that it is red and second in that it has three states, OFF, HALF BRIGHT, and FULL BRIGHT. The significance of each state is:

**OFF:** Both the CELL key and the CELL ENABLE switch are OFF, OR the CELL key is OFF and the CELL ENABLE switch is ON.

**HALF BRIGHT:** The CELL key is ON but the CELL ENABLE switch is OFF.

**FULL BRIGHT:** Both the CELL key and the CELL ENABLE switch are ON. The cell is ON only when the light is at full brightness.

Users might note that there is also a Cell switch at the electrometer. This switch has two positions, DUMMY, and EXT. Its function is to determine whether the Model 273A is controlling with respect to the external cell or with respect to an internal (dummy cell) resistor.

**Note:** This switch provides no protection in either position. The cable connections can be handled safely only when the front panel cell disable switch is set to OFF.

Note that the Model 273A is controlled even when the cell isn't ON, that is, having the cell OFF does not cause an overload condition.

It might be noted that, in the context of this discussion, Cell ON and Cell OFF refer to the path from the Control Amplifier output to the Counter Electrode cable lead. The red indicator does not respond to the status of the actual counter electrode connection. That connection could be secure or faulty and it would have no effect on the red indicator, which responds only to the Cell ON switch, the CELL ENABLE switch, and governing software. However, if the Cell is ON and there is no connection to the counter electrode, the effect will be the same as if the cell had infinite resistance and an E OVERLOAD will occur.

#### 2.5. Defects and Abnormal Stresses

Whenever it is likely that the protection provided by the connection to earth ground has been impaired, do not use the instrument and secure it so others cannot use it. The protection is likely to be impaired if, for example, the apparatus:

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

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

## 2.6. Ventilation

The Model 273A incorporates forced-air ventilation to maintain a safe operating temperature. Thus it is necessary to allow some free space (minimum 10 cm) behind the instrument so that adequate air circulation can occur. Moreover, there must be adequate circulation between the space behind 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 273A is cabinet or rack mounted, some additional effort to assure adequate ventilation may be required. Ambient Temperature: From 10°C to 40°C. The instrument shall operate from 10°C to 40°C but may not meet some temperature related specifications. The instrument shall operate from 20°C to 30°C and meet all its specifications over this range. The ambient temperature should not exceed 45° C (113° F).

#### 2.7. This Apparatus as a Source of Radio Frequency Interference

In a typical application, it is unlikely that this apparatus will act as a source of noticeable radio frequency interference. However, when operated near particularly sensitive equipment, interference emanating from this apparatus 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 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 metal braided or solid strap. (A solid strap does a better job but is more clumsy. Copper, aluminum, or brass are the recommended materials).

Because the Model 273A 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 apparatus 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 fitter 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 dose 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.

# 2.8. Transient Sensitivity

Generally speaking, the design and construction techniques used in equipment manufactured by Princeton Applied Research 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 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. Suitable devices are commercially available.

# 3. CHARACTERISTICS

# 3.1. Specifications

The following specifications apply at the nominal line voltage  $\pm$  10% and at a temperature of 25°C (77°F) unless otherwise stated.

#### 3.1.1. Power Amplifier

- 1. Compliance Voltage: > ± 100 V
- 2. Maximum Output Current: > ± 1.0 A
- 3. Slew Rate: 10  $V/\mu$ s (high speed)
- 4. Bandwidth, Open Loop, Unity Gain: >2.5 MHz
- 5. Voltage Temperature Stability. <50 μV/ °C

#### 3.1.2. Differential Electrometer

- 1. Input Impedance: >10 $^{\circ}\Omega$  in parallel with <50 pF
- 2. Input Bias Current: <20 pA at 25°C
- 3. Maximum Input Voltage

Differential: ± 10 V

Reference Input: ± 11 V

- 4. Common Mode Rejection
  - > 80 dB from dc to 1 kHz
  - > 40 dB at 100 kHz
- Bandwidth

Small Signal: > 4 MHz

Full Signal: > 400 kHz

- 6. Offset Voltage: <10  $\mu$ v
- 7. Offset Temperature Stability-. < 10  $\mu$ V/°C

#### 3.1.3. Current Measurement

- 1. Ranges: 8 decades, 1 A to 100 nA
- Accuracy (dc) at Monitor

10  $\mu$ A to 1 A: Better than 0.2% of range

100 nA and 1  $\mu$ A Ranges: Better than 0.5% of range ±5 nA max ( ± 1 nA typical)

3. Frequency Response (small signal)

1 mA Range: -3 dB at > 1 MHz, 1 k $\Omega$  source impedance 10  $\mu$ A Range: -3 dB at > 75 kHz, 100 k $\Omega$  source impedance

#### 3.1.4. Potential/Current Control

1. Digital/Analog Converters (DAC's)

Bias DAC

Resolution: 14 bits

Range: ±8 V (potentiostat)

±200% of full-scale current (galvanostat)

Modulation DAC Resolution: 14 bits

Range (Poten.):  $\pm 2$  V,  $\pm 0.2$  V, and  $\pm 0.02$  V

Range (Galvan.): ±200%, ±20.00%, and ±2.000% of full-scale current.

2. Accuracy

Applied Potential: 0.2% of reading ±2 mV Applied Current: 0.2% of full-scale current

#### 3.1.5. IR Compensation

1. Positive Feedback

Digitally Controlled Range: 1/Current Range (0 to 2 times the Current Range Resistor) Resolution: 0.05% of Current Range Resistor

2. Current Interrupt

Digital Potential Error Correction: 12 bit DAC

Total Interruption Time:  $< 200 \mu s$ 

Switching Time, ON/OFF:  $< 1 \mu s$  (1 k $\Omega$  resistive cell)

#### 3.1.6. System

1. Rise Time (10% to 90% on high-speed setting)

No Load: < 750 ns 1Ω, 1 A:  $< 3 \mu$ s 10 kΩ, 100  $\mu$ A:  $< 2 \mu$ s

2. Noise and Ripple: typically  $< 25 \mu V$  rms referred to external input

#### 3.1.7. Computer Interfaces

- 1. RS-232C
- 2. IEEE-488 (GPIB)

The instrument recognizes more than 100 different commands for control from a remote computer via the IEEE-488 or RS-232C interface. The *Model 273A Remote-Programming Command Handbook* describes these commands and provides detailed explanations of GPIB and RS-232C communications, including rear-panel switch settings and communications protocols.

#### 3.1.8. Weight

31 kg (68 lb)

#### 3.1.9. Size

48 cm W  $\times$  30 cm H  $\times$  51 cm D (19" W  $\times$  12" H  $\times$  20" D)

#### 3.1.10. Power Requirements

100-130 V or 200-260 V, 50-60 Hz, 350 watts maximum

## 3.1.11. Rack Mounting

The Model 273A may be mounted in a standard 19 inch (47.5 cm) rack assembly. If rack-mounted, the Model 273A must be supported to avoid excessive stressing of the front panel. Appendix B provides instructions for rackmounting.

#### 3.1.12. Battery Backup

A battery-powered parameter backup system is provided. This system assures that all parameters retain the values in effect at the end of the previous operating session. In other words, shutting the Model 273A off does not cause the default values to be restored. They can, however, be restored by reinitializing the system via FUNCTION 10. The use of FUNCTION 10 is described in Section 5.2.4.

Another way of restoring the default parameters is to press and hold in the LOCAL key as the Model 273A is being powered up. The LOCAL key must be held in until the message "SYSTEM REINITIALIZED" appears on the front-panel display.

#### 3.1.13. Front-Panel Connectors

 EXT INPUT: ± 10 V analog input. Potential applied is summed with that set at front panel or applied via digital interface. Input impedance 100 kΩ.

- E MONITOR: ± 10 V analog output with output impedance of 1 kΩ. This is the potential of the working electrode with respect to the reference electrode. IR compensation, if active, will affect the E MONITOR output level This same output is provided at the rear-panel RECORDER INTERFACE connector.
- 3. I MONITOR: I/E Converter Output.  $\pm$  1 V for full-scale current; 2 V maximum. Output impedance:  $1k\Omega$ .
- 4. OUTPUT: Analog output that tracks current (linear or log) or charge (coulombs). Output impedance is 1 k $\Omega$ .

Linear: Tracks I MONITOR output; 1 V for full-scale current; 2 V maximum.

Log: 1 V per decade;  $\pm$  10 V maximum. A current equal to the Log Reference Range (selected by Function 12) gives 0 V out.

Coulombs: ± 10 V. Initial sensitivity is one full-scale current for one second gives 10 V out. Sensitivity decreases by factor of ten at 10 V. Sensitivity ranging repeats as required to accommodate total charge. Bipolar.

#### 3.1.14. Rear-Panel Connectors

- 1. CELL INTERFACE: Connections to cell are made with provided cable via this connector.
- 2. POWER AMP MONITOR: Amplifier output divided by ten.  $\pm$  100 V range of amplifier output gives  $\pm$ 10 V POWER AMP MONITOR output. Output impedance is 1 k $\Omega$ .
- 3. ELECTROMETER MONITOR: Output of the differential electrometer. Not corrected for IR drop (unlike front-panel E MONITOR output). Output impedance is 1 k $\Omega$ .
- 4. AUXILIARY INTERFACE: Provides triggers and Model 303A control signals. Note that the Model 303A is not cabled directly to this connector. It rather is cabled to the Model 307 Interface, which in turn is cabled to the AUXILIARY INTERFACE connector.
- 5. AUX A/D INPUT:  $\pm 10$  V, high-impedance, analog input (useful for galvanic corrosion, spectro-electrochemical, and other measurements). Input impedance:  $> 10^9 \Omega$ .
- 6. RECORDER INTERFACE: 15-pin connector provides Y output (identical to front-panel OUTPUT signal), X output (identical to front-panel E MONITOR signal), contact closure lines for pen up/down control, and ground.
- 7. RS-232C INTERFACE: Serial port for control of the Model 273A from an external computer. Associated switch assembly sets RS-232C parameters (exception: Terminator, <CR> or <CRLF>, is set by switch in the GPIB switch assembly). **Note:** The maximum potential that can be applied to this port is ± 15 V. The *Model 273A Remote-Programming Command Handbook* describes switch settings and communications protocols.
- 8. IEEE-488/GPIB INTERFACE: Parallel port for control of the Model 273A from an external computer. Associated switch assembly sets GPIB Address, Terminator, and status of Test Echo function. The *Model 273A Remote-Programming Command Handbook* describes switch settings and communications protocols.
- 9. AC I OUTPUT (used only in units equipped with the 273A/92 Electrochemical Impedance Interface option): Outputs the ac current signal from the test cell. The signal is controlled by commands from an external computer via the GPIB or RS-232C port.

- 10. AC E OUTPUT (used only in units equipped with the 273A/92 Electrochemical Impedance Interface option): Outputs the ac voltage signal from the test cell. The signal is controlled by commands from an external computer via the GPIB or RS-232C port.
- 11. MULTIPLEXED OUTPUT (used only in units equipped with the 273A/92 Electrochemical Impedance Interface option): Either a current or voltage signal can be output from this connector as determined by commands from an external computer via the GPIB or RS-232C port. Note: This output is intended for use with Princeton Applied Research electrochemical impedance systems.
- 12. AC INPUT (used only in units equipped with the 273A/92 Electrochemical Impedance Interface option): This connector accepts a sine wave input from an external oscillator, such as that supplied by a Princeton Applied Research lock-in amplifier. The nominal frequency range is 50  $\mu$ Hz to 100 kHz, although this is dependent on loading. Input voltage must be no greater than 5 volts rms. This signal can be used to modulate the signal sent by the Model 273A to the cell, and can be attenuated as desired. It is controlled by commands from an external computer via the GPIB or RS-232C port.

#### 3.1.15. 273A/92 Electrochemical Impedance Interface Option

The 273A/92 Electrochemical Impedance Interface option (previously called the AC Interface option) is intended primarily for use with Princeton Applied Research electrochemical impedance systems, but can also be used for ac voltammetry applications. This option is designed to allow superposition of an externally generated ac excitation signal on the dc signal generated by the Model 273A. The *Model 273A Remote Programming Command Handbook* describes the computer commands used with the Model 273A/92 option. Section 3.2.10 of this chapter describes how it works.

#### 3.1.16. Accessories

- MODEL 407A INTERFACE: This accessory allows the Model 273A to be used in conjunction
  with the Model 303A Static Mercury Drop Electrode. This adapter requires two cables to
  make the necessary connections. One cable connects to the Model 273A AUXILIARY
  INTERFACE connector and the other to the Model 303A INPUT connector. The Model 407A
  requires +24 V at 1 A. A suitable power supply is included with it.
- 2. MODELS RE0150 and RE0151 RECORDERS: These recorders are well suited to use with the Model 273A and a custom cable is available to simplify the necessary interconnections. Both recorders use 11 x 17 inch paper. The principal difference between them is that the RE0151 incorporates a time base.
- 3. In addition, a variety of cells, electrodes, and other items are available as described in the Princeton Applied Research Electrochemical Accessories Catalog.

#### 3.1.17. Polarity Convention

The Model 273A 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. Negative current is anodic, that is, a current is defined as negative if oxidation is taking place. A full description of the polarity convention used is given in 1.1.4.

#### 3.1.18. Metric Hardware

Equipment manufactured by Princeton Applied Research uses American Standard and metric fasteners and related hardware. For this reason an electronic technician servicing or aligning the apparatus should make a special effort to keep track of any hardware removed from the chassis, the cable connectors, and attached equipment.

The differences between metric and American Standard hardware are not always easily recognized. As an aid, metric hardware is marked with a yellow chromate finish or, in the instance of metric screws, with a yellow locking pad applied to the threads.

# 3.2. Internal Organization

#### 3.2.1. Introduction

Although the Model 273A is a complex instrument, its basic organization is straightforward as depicted in Fig. 3. The following paragraphs discuss the Model 273A, as represented in the figure, in some detail. The symbols used in Fig. 3 are, for the most part, conventional, and require no description. However, it should be noted that the many switches drawn inside rectangular boxes are digitally controlled switches, and not under direct mechanical control. The gains and scaling of internal signals are omitted for the sake of clarity.

#### 3.2.2. Bus

Referring to Fig. 3, notice the bus that interconnects all of the major circuit blocks. All of these blocks communicate with each other and with the controlling microprocessor via this bus. This is true both for control via the front panel and for control from an external computer via the GPIB or RS-232C port.

## 3.2.3. Applied Potentials and Currents

Note the Control Amplifier, located near the center of the figure. As shown, many different signals are applied to the input of this amplifier. However, only three of these "inputs" are sources of applied (control) potential or current, that is, user-controlled sources that are summed together and that directly set the current or potential applied to the cell. Those three are:

- 1. BIAS DAC (Digital-to-Analog Converter), the output of which is scaled to provide either ±8 V in applied E Mode, or ±2X full scale current in APPLIED I mode.
- 2. MOD DAC, the output of which, also ± 10 V, is scaled to ±2 V, ±20 mV, ±20 mV in APPLIED E mode operation, or ±2 × full scale in APPLIED I mode operation.
- 3. EXT INPUT. The voltage applied to the input causes an equal voltage to be applied to the cell in CONTROL E mode. In CONTROL 1 operation, 1 V applied to this input causes one full scale of current to be applied to the cell.

As previously mentioned, these sources are summed. However, the sum must not exceed ±10 V in APPLIED E operation or ±2 times full scale current up to 1 A maximum in APPLIED I operation. Each of these three inputs can be disabled without changing the values at the DAC's by means of the associated CMOS switches (switches depicted in rectangular boxes). The switches for the two DAC's are shown to the right of the Scaling Circuits. The switch for the External Input is shown to the right of the Buffer Amplifier. Note that the switches for the DAC's cannot be accessed from the front panel or via the command set. The External Input switch, on the other hand, is controllable via the command set (EXT command). The *Model 273A Remote-Programming Command Handbook* describes the computer command set.

#### 3.2.4. Analog Circuits

The analog section of the Model *273A* comprises three operational amplifiers. They are: (1) the Control Amplifier, (2) the Electrometer, and (3) the Buffer Amplifier that drives the I/E MONITOR connector. Note that this potentiostat does NOT incorporate a grounded-potential working electrode; the working electrode can be off ground by as much as 2 V. The 2 V occurs because of the potential drop across the current sensing resistor.

#### **Buffer Amplifier; I Measurement Circuit:**

Referring to Fig. 3, note the Current Range Relays in the box to the right of the Buffer Amplifier that drives the I/E MONITOR connector. These computer-controlled relays select any one of the

eight Current Range Resistors. Values from  $1\Omega$  to  $10M\Omega$  can be selected, with the result that a full-scale current (1 A to 100 nA) will cause a drop of 1 V. Since currents as high as two times full scale are permitted on all but the 1 A range, drops as high as 2 V can occur. The circuit is configured such that the Buffer Amplifier makes a four-terminal measurement of the voltage drop across the Current Measurement Resistor, thereby assuring that the current measurement is independent of cable and switch contact resistance in the switch and in the connecting cell interface.

#### **Differential Electrometer:**

The Differential Electrometer is located in a remote enclosure to minimize lead capacitance. This differential electrometer allows accurate potential control independent of lead and contact resistance. The inverting (-) input of the electrometer connects to the working electrode ("top" of the current sensing resistor), either directly, or via the sense lead if the remote sense is enabled by removing the sense jumper and running a separate sense lead to the cell. The non-inverting (+) input of the electrometer is normally connected to the reference electrode. Thus the output of the electrometer is the potential of the reference electrode versus the working electrode, that is, the parameter to be controlled in CONTROL E operation and measured in the CONTROL I operation.

#### **Control Amplifier (Power Amplifier):**

This circuit is basically a  $\pm$  100 V,  $\pm$  1 A power operational amplifier. The various input signals are applied through resistors (not shown) and summed together at the input of the Control Amplifier. The output of the Control Amplifier is driven to whatever voltage is required to cause the feedback signal to be equal and opposite to the summed input signals. The feedback signal is an analog of either the cell potential or current, as determined by the E or I feedback switches according to the operating mode. In the CONTROL E mode, the feedback signal is taken from the electrometer. In the CONTROL I mode, it is taken from the current measurement circuit. The Control Amplifier has computer-controlled frequency response, shown in the block labeled STABILITY SELECTION. In both CONTROL E and CONTROL I operation, the HIGH STAB mode limits the bandwidth of the Control Amplifier by changing the capacitor in its feedback loop. This is done to assure stability on most routinely encountered cells and experimental situations. The Control Amplifier is faster without these frequency adjusting components but may oscillate under certain conditions.

Note that a Power Amp Monitor point is provided at the rear panel. To eliminate any shock hazard, the amplifier's output voltage is internally divided by ten prior to being brought out to the Monitor point.

#### 3.2.5. Cell Switch and Relay

Referring to Fig. 3, note the Cell Switch and Cell Relay shown in series between the output of the Control Amplifier and the counter electrode connection (red clip lead). Even though the block diagram shows two switches in series, there are actually three of them. The first is the manually operated CELL ENABLE Switch provided at the front panel as a fail-safe means of rendering the red clip lead harmless. The second switch is a high-speed solid-state device and cell relay capable of connecting and disconnecting the cell in less than one microsecond.

Although primarily intended to implement Current Interrupt IR Compensation, it is also used for connecting and disconnecting the cell to provide "glitchless" transitions from Cell ON to Cell OFF and vice versa. However, there is finite leakage across this solid-state switch when the cell is OFF. Hence the third switch, the Cell Relay shown in the figure. Although this relay is much slower than the solid-state switch, its low leakage assures a very high degree of cell isolation. Both the manually operated Cell Enable switch and the computer controlled solid-state switches (relay and solid-state switch) provide a feedback path for the Control Amplifier when the cell isn't connected. The gain in this path is nominally X1 (solid state switch) or X1.5 (relay). If one volt is applied, the Control Amplifier output will be 1 V or 1.5 V, as appropriate.

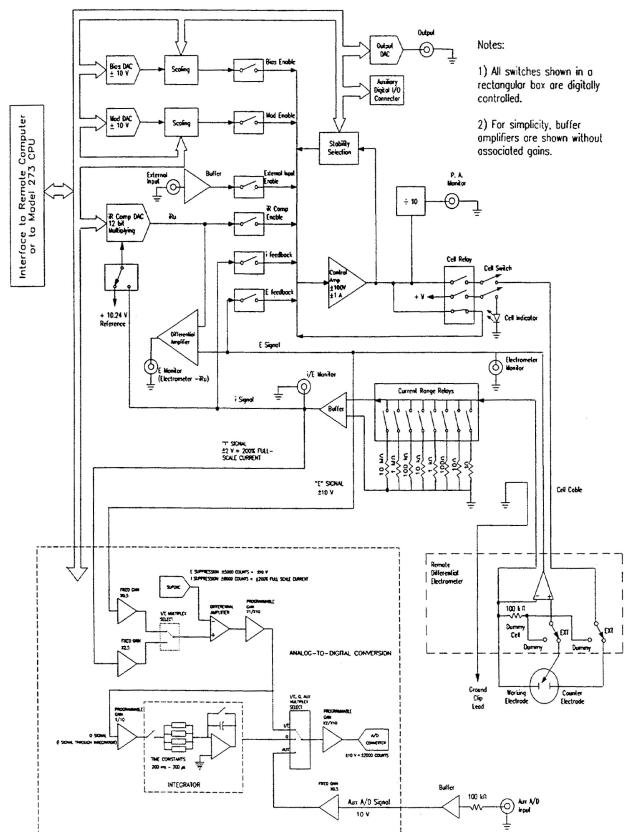


Fig. 3. Model 273A Simplified Block Diagram.

#### 3.2.6. IR Compensation

There are two modes of IR Compensation, Current Interrupt and Positive Feedback. When Current Interrupt is enabled, the IR COMP DAC applies the correction voltage derived as a fraction of the 10.24 V reference. Note that the switch shown beneath the DAC is shown in the "fixed" reference position, in which the reference input to the DAC is the aforementioned +10.24V level.

When Positive Feedback IR Compensation is enabled, a fraction of the voltage developed by the current measurement circuit is fed back as an input to the Control Amplifier through the DAC. Thus, in this mode, the switch beneath the DAC connects the output of the Current Measurement circuit to the DAC's reference input. The front-panel E MONITOR output is the electrometer signal corrected for the amount of iR compensation as shown in the block diagram.

## 3.2.7. Integration

The integration circuitry on the IR Integrator board provides for *analog* integration of current for short timescale coulometry experiments (50  $\mu$ s  $\leq$  TMB  $\leq$  40 ms). The effective time constant for measurements ranges from 40 Ns to 40 ms and is controlled by the ITC command. The effective time constant combines with the current range to determine the full-scale output of the integrator. The GIGAIN command further affects the effective time constants. See the ITC command in the *Model 273A Remote Programming Command Handbook* for more information.

Longer-time scale coulometry experiments (TMB  $\leq$  40 ms) with slowly varying currents may be performed using SIE 1 to collect current directly followed by numeric integration using either INT or IINT commands (see the *Command Handbook*).

#### 3.2.8. Analog to Digital

The Analog-to-Digital conversion section of the instrument has the ability to digitize any one of four applied signals:

- 1. The output of the Current Measurement Circuit (I Signal in the block diagram).
- 2. The I signal after processing by the iR Integrator circuit (Q Signal in the block diagram).
- 3. The Electrometer Output (E Signal in the block diagram).
- 4. An Auxiliary A/D Input (Aux A/D Signal in the block diagram).

The A/D section incorporates a low-pass filter for the I signal (only), a computer selectable gain-of-ten for both the E and I signals, and a computer-selectable additional gain of five for all four signals (E, I, AUX, and Q). The X2 - X10 buffer's output is applied to a Sample-and-Hold circuit, which in turn drives a 12 bit A/D Converter that connects to the bus. The A/D Converter, prior to the gains, has a nominal full-scale range of  $\pm$  10 V in MEASURE E,  $\pm$ 2 times full-scale current in MEASURE I, and  $\pm$ 10 V in AUX A/D. The gain settings made further on are always relative to these base values.

Both the E and I signals can be offset by the SUPDAC before applying the gains to the signal. This allows the measurement of small differences on top of large dc values.

#### 3.2.9. OUTPUT and AUX DIGITAL Connector

The OUTPUT provides a voltage proportional to the current or to the computer-calculated quantities of charge and log current. This is also the signal applied to the Y-axis at the RECORDER INTERFACE connector.

The AUX DIG I/O interface is used for experimental control and is compatible with TTL as described in Appendix A.

#### 3.2.10. 273A/92 Electrochemical Impedance Interface Option

The 273A/92 Electrochemical Impedance Interface option (previously called the AC Interface option) is intended primarily for use with Princeton Applied Research electrochemical impedance systems, but can also be used for ac voltammetry applications. This option is designed to allow superposition of an externally generated ac excitation signal on the dc signal generated by the Model 273A.

The externally generated ac signal is input at the AC INPUT connector and is combined with the signal generated by the Model 273A before it is applied to the cell (see Fig. 4). The /92 option board has three output connectors (AC I OUTPUT, AC E OUTPUT, and MULTIPLEXED OUTPUT. Software commands control both the mode of output (I, E, or multiplexed) as well as the type of signal applied (full ac + dc signal or ac signal only). Multiplexing is accomplished by alternating the operand of the MIE command between "1" and "2" on successive iterations. The *Model 273A Remote-Programming Command Handbook* describes the computer commands used with the Model 273A/92 option.

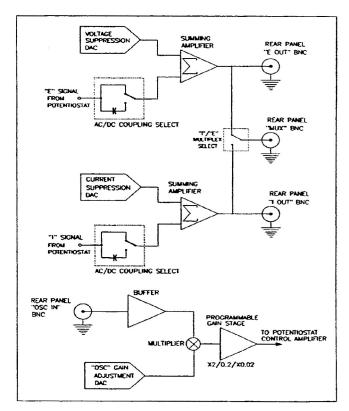


Fig. 4. Simplified Block Diagram of Model 273A/92 Electrochemical Impedance Interface Option.

# 4. INITIAL CHECKS

#### 4.1. Introduction

The following procedure is provided to facilitate initial performance checking of the Model 273A. Perform this procedure after you have inspected the instrument for obvious shipping damage. Any damage noted should be reported to the carrier and to Princeton Applied Research. Be sure to save the shipping container for inspection by the carrier.

If you have not used a Model 273A or 273 before, this procedure also will help acquaint you with the operation of the instrument.

The Model 273A is checked using a resistor as the cell. These checks are adequate for determining that the instrument is functioning normally.

Note that these procedures are not intended to demonstrate that the instrument meets specifications, but rather to demonstrate that it has arrived in good working order. Each Model 273A receives a careful checkout before leaving the factory, and ordinarily, if no shipping damage has occurred, it 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. Required Equipment

Other than the Model 273A to be checked and its electrometer, no other equipment is required.

## 4.3. Setup

- 1. Set the Model 273A with electrometer on a lab bench. Or, if you prefer, mount it in a standard 19-in. (47.5 cm) rack assembly. Instructions for rack mounting are provided in Appendix B.
- 2. Set the Model 273A's POWER ON/OFF switch to the OFF position.
- 3. Set the Model 273A CELL ENABLE pushbutton switch to OFF.
- 4. Conned the Electrometer/Cell cable to the CELL INTERFACE connector at the rear of the Model 273A. Bring the electrometer around to the side of the Model 273A for easy access.
- 5. Locate the shorting plug supplied with the electrometer. Plug it into the SENSE (gray) and WORKING (green) electrometer jacks, shorting them together.
- Set the switch located on the Electrometer to the DUMMY position. (In this position an
  internal resistor acts as the cell.) **Note:** This is a locking switch. To change its setting the
  toggle must be pulled out a millimeter or two as required to disengage the locking
  mechanism.
- 7. Note the GROUND, COUNTER ELECTRODE, and WORKING ELECTRODE leads that emerge from the electrometer. In the following checks, take care that the alligator clips at the end of the leads don't short to each other or to any electrically conducting material.

**Warning:** Although the external cell is disconnected when the electrometer switch is set to DUMMY, the potential on the Counter-Electrode lead can be as high as 100 V and must be considered dangerous in case the switch is accidentally set to EXT.

Plug the Model 273A into a suitable source of ac power. (See Chapter 2 for line voltage selection and safety information.)

- 9. Set the 273A's POWER ON/OFF switch to ON.
- 10. Reinitialize the system by pressing the following sequence of keys:

#### [FUNCTION] [1] (0] [YES] [ENTER]

#### 4.4. Procedure

The following steps program the Model 273A to do a controlled-potential experiment on the  $100 k\Omega$  dummy cell resistor located in the electrometer housing. 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 to 10  $\mu$ A, and then decreasing to zero again. A voltage corresponding to this current will appear at both the I MONITOR connector and at the OUTPUT connector. With a selected current range of 10  $\mu$ A, the voltage at these connectors will start at 0 V, increase to 1 V, tracking the scan, and then return to 0 V. The current in the resistor will be displayed throughout the experiment.

**Note:** The dummy cell resistor has a 1% tolerance rating. As a result, the current indications can be at least 1% off with respect to the values indicated in this procedure. Note also that residual drift or noise in the Model 273A itself can contribute a small additional

- 1. Note the Model 273A display. There are two information lines. The measured cell current and potential are displayed on the upper line. The lower line displays various messages.
- 2. Press the [POTENTIOSTAT] key. The associated indicator should light, indicating that the 273A is in the controlled-potential mode.
- 3. Pres [E/I 1]. The display message line will indicate that the E/I 1 is to be entered. Then key.

#### [0] [ENTER]

entering 0 V as the programmed starting potential for the scan to be performed.

4. Press [DELAY 1]. The display message line will indicate that DELAY 1 is to be entered. After that, key.

entering 10 s as the time for which E/I 1 (0 V) will be applied before beginning the scan.

Note the message at the end of the lower display line. It should indicate that the delay will be performed with the cell off. If it indicates the CELL ON state, press the Scan Setup CELL OFF key to establish the desired CELL-OFF delay.

Press [SCAN 1]. The display message line will indicate that SCAN 1 is to be entered. Next key.

#### [2] [0] [ENTER]

entering 20 mV/s as the programmed scan rate for the first leg of the scan.

6. Press [E/I 2]. Then key:

#### [1] [.] [0] [0] [0] [ENTER]

entering 1.000 V as the scan vertex potential.

7. Press [DELAY 2]. Then key:

#### [1] [0] [SEC] [ENTER]

establishing 10 seconds as the time for which the vertex potential will be applied.

8. Press [SCAN 2]. Then key:

#### [2] [0] [ENTER]

establishing a scan rate of 20 mV/s for the second leg of the scan as well.

9. Press [E/I 3]. Then key:

#### [0] [ENTER]

establishing the scan-end potential at 0 V, which was also the starting potential.

- 10. Locate the Cycle Select key (second key from the bottom in the CONTROL group). Press this key as required to light the SINGLE CYCLE indicator.
- 11. Locate [ $\uparrow \uparrow$ ] and [ $\downarrow \downarrow$ ] keys in the CURRENT RANGE group. Operate these keys as necessary to light the 10  $\mu$ A range indicator. *AUTO must be OFF (adjacent light will be extinguished.)*
- 12. Locate the OUTPUT SELECT key (bottom key of OUTPUT group). Operate this key as necessary to light the LINEAR indicator.
- 13. Set the CELL ENABLE switch to the ON position.
- 14. Press the CELL ON key, lighting the associated indicator.
- 15. Press the ENTER key as many times as are required to blank the bottom line of the display.

The Model 273A is now ready to run the experiment. The programmed sequence will be initiated in the following step.

- 16. Press START. The following should be observed. **Note:** START can be pressed at any time to restart the check sequence.
  - a. The E/I 1 indicator (scan status display at upper left-hand corner of the front panel) will flash once, indicating that the initial potential has been applied. This potential (0 V) and the resulting current (0 A) will be indicated on the upper display line. **Note:** There is always some 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 10  $\mu$ A; 1% of that is 100 nA). Potential indications should be within 10 mV of the indicated value.
  - b. The DELAY 1 indicator (scan status display) will light and remain lighted for 10 s, the programmed delay interval. The displayed potential and current will remain constant.

- c. The SCAN 1 indicator (scan status display) will light and remain lighted for the 50 s it takes to complete the first leg of the scan at 20 mV/s. As the scan advances, the display will indicate the increasing potential and current.
- d. The E/I 2 indicator (scan status display) will flash once, indicating that the scan has reached 1 V, the programmed vertex potential.
- e. The DELAY 2 indicator (scan status display) will light and remain lighted for 10 s, the programmed delay interval. The displayed potential (1 V) and current (10  $\mu$ A) will remain constant.
- f. The SCAN 2 indicator (scan status display) will light and remain lighted for the 50 s it takes to complete the second leg of the scan at 20 mV/s. As the scan advances, the display will indicate the decreasing potential and current.
- g. The E/I 3 indicator will light and remain lighted, indicating that the scan is complete.
- 17. Set the CELL ENABLE switch to OFF.
- 18. Press the CELL ON/OFF key, establishing the Cell OFF state. The associated indicator light should extinguish.
- 19. Set the Model 273A POWER ON/OFF switch to OFF.

This completes the initial checks. If the indicated behavior was observed, the user can be reasonably confident that all circuits up to and including the electrometer are functioning normally.

# 5. OPERATING INSTRUCTIONS

#### 5.1. Introduction

Although the Model 273A is a complex, microprocessorcontrolled analytical instrument, its operation is, nevertheless, straightforward. The instrument's front panel has been carefully designed with attention to humanengineering considerations. Its two-line alphanumeric display continuously displays the potential and current on one line, while guiding the operator through the operating sequence via messages displayed on the second. A special Scan Status Display is provided that continuously indicates the stage in progress of an ongoing scan. Front-panel indicator lights allow the user to ascertain the setup at a glance.

A battery-powered parameter backup system retains all selections and parameter values. When the 273A is powered up, all selections and parameter values are as they were at the end of the previous operating session. The default values can, however, be restored by reinitializing the system via FUNCTION 10. The use of FUNCTION 10 is described in Section 5.2.4.

Another way to restore the default values is by pressing the LOCAL key when powering up. The key must be held in until the message "SYSTEM REINITIALIZED" appears on the front-panel display.

No effort has been spared to make the Model 273A an easy instrument to operate. Nevertheless, information beyond that provided on the front panel is required for optimum performance in many situations. This chapter of the manual addresses those needs.

Instructions for operating the unit remotely via either the RS-232C or GPIB (IEEE-488) interface port are given in the separately bound Model 273A Remote-Programming Command Handbook.

#### 5.2. Front Panel

The Model 273A's front panel (Figure 5-1) is divided into clearly delineated functional areas. The SCAN STATUS DISPLAY, OVERLOAD INDICATORS, and ALPHANUMERIC DISPLAYS are located at the top of the panel. The key/indicator areas, SCAN SETUP, CONTROL, MODE, CELL, INPUT, FILTER, IR COMPENSATION, CURRENT RANGE, OUTPUT, and INTERFACE are located beneath them. Each area's features are discussed in some detail in the following paragraphs.

# 5.2.1. Alphanumeric Display

The Model 273A incorporates an LCD alphanumeric display able to show two lines (40 characters each) of both upper- and lower-case characters. The measured potential and current are continuously displayed on the upper line. Messages and parameter values appropriate to the operation being performed are displayed on the lower line.

For example, when a setup parameter key is pressed, the previously set value is displayed, together with the message, ENTER XXX (XXX signifies the parameter). The new value can then be keyed and ENTER pressed, after which the new value will be displayed. Pressing ENTER again clears the new value from the display so that it can return to the normal bottom line mode, in which the bottom display line can scroll through several possible display options, advancing once each time the ENTER key is pressed. The choices are:

- 1. Second Display Line Blank.
- 2. Charge in coulombs.
- 3. Log of I if LOG is enabled.

- 4. Status of the scan, such as:
  - a. Delay Time Remaining (Delay specified in seconds).
  - b. Time Remaining Until Drift Measurement Can be Made (Delay specified in mV/s). Alternatively, measured drift rate if measurement has taken place.
  - c. Stop Scan.
  - d. E Applied or I Applied, according to the selected mode.

Error messages can also be displayed on the bottom line. These include front-panel Error Messages, which can be cleared by pressing any key, and Remote Error Messages, which only occur when controlling the Model 273A from a remote computer. Remote Error Messages are in two parts, the Error #, and then the Command that caused the failure. Remote Error Messages can only be cleared by taking appropriate action at the remote computer or cycling the power. The bottom line display can also be used to scroll through the available Overrides (see Section 5.2.4) using the up and down arrow keys. This capability becomes available once one of the overrides has been called up for display.

To the right of the display is a screwdriver adjustment that allows the display's angle to be adjusted for optimum readability.

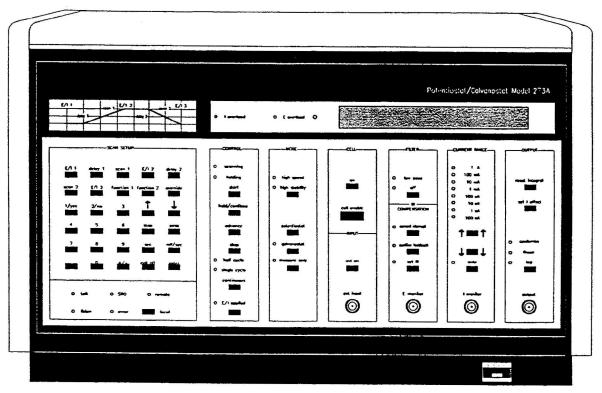


Fig. 5. Model 273A Front Panel.

#### 5.2.2. I Overload and E Overload

These two indicators are located between the Scan Status and Alphanumeric Displays. The I OVERLOAD indicator lights if the cell current exceeds two times the selected Current Range. Because it is impossible to reach currents of 2 A, this lamp cannot light when the current range is "1 A." However, currents greater than 1 A will cause the E OVERLOAD indicator to light.

The E OVERLOAD indicator lights if the control amplifier output is at its limit (I > 1A or  $E_{control}$  > 100 V) and the control loop is not controlling the cell potential or current. 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.

#### 5.2.3. Scan Status Display

The Scan Status Display, located at the upper left of the front panel, consists of a symbolized scan waveform divided into sections marked by LED's (light emitting diodes) that correspond to the different stages of a scan. As a scan progresses, the LED corresponding to the scan stage in progress lights, informing the user of the scan's status at a glance. There are seven LED's on the Scan Status Display waveform. A brief discussion of each follows.

- 1. E/I 1: When a scan is initiated (START key), this indicator lights for 1/4 second to advise the operator that the scan sequence has started. This may or may not correspond to actually applying E/I 1 to the cell. For example, if the scan program includes a DELAY 1 interval, the delay could be done with the cell ON or OFF. If the delay is done with the cell ON, the lighting of the E/I 1 indicator marks the moment when E/I 1 is applied. If the delay is done with the cell OFF, the lighting of the E/I 1 indicator means that the programmed scan sequence has been initiated and nothing more.
- DELAY 1: Scans typically (but not necessarily) begin with a delay interval, and the DELAY 1 indicator lights while the delay is in progress. If Delay 1 is PASS'ed, the DELAY 1 indicator doesn't light. If the Delay 1 interval is 1/4 second or less, the indicator lights for 1/4 second. See the description of the E/I 1 key for a discussion of the Delay Options.
- 3. SCAN 1: This indicator lights as soon as the scan waveform is applied and remains lighted while it is in progress. If the scan is 1/4 second or less, the light remains on for 1/4 second. If a STEP is applied instead of a ramp, the SCAN 1 indicator doesn't light at all.
- 4. E/I 2: This indicator lights for 1/4 second when the scan (or step) reaches the vertex potential (current), E/I 2.
- 5. DELAY 2: This indicator lights during the Delay 2 interval. If this second delay is PASS'ed, DELAY 2 doesn't light at all. If the delay lasts 1/4 second or less, the light remains on for 1/4 second.
- SCAN 2: This indicator lights during the second leg of the scan. If the scan is 1/4 second or less, the ight remains on for 1/4 second. If a STEP is applied instead of a ramp, SCAN 2 doesn't light.
- 7. E/I 3: This indicator lights for 1/4 second at the end of the scan, indicating that the programmed sequence has been completed.

**Note:** If the entire scan is too quick to be displayed in real time by the LED's, the LED's that correspond to the selected states light in sequence to show the operations performed. The timing of this display sequence is not linked with that of the scan.

#### 5.2.4. SCAN SETUP Group

This set of thirty keys is used to set the parameter values and sequences for experiments performed with the Model 273A. When one parameter key is pressed, the set value is displayed (second line of display), together with the message ENTER XXX, where XXX is the parameter of interest. To change a parameter, simply key the new value and press [ENTER]. The new value replaces the previous one and is displayed. **Note:** Throughout the following text, brackets are used to denote a key to be pressed. For example, [ENTER] designates the Enter key.

To simply check a parameter value, press the corresponding setup key and note the displayed value. Press any other key to leave the operation. [ENTER] is a good choice because its only effect will be to reinstall the value already in force.

When a setup parameter is displayed, it can be changed by pressing the Setup Up Arrow key  $[\uparrow]$ , which increases a parameter's value, or the Setup Down Arrow key  $[\downarrow]$ , which decreases it. It is not necessary to press the [ENTER] key to implement changes made with the arrow keys. This technique is recommended for making small value changes. These keys can also be used to change parameter values during an actual scan (see discussion of  $[\uparrow]$  and  $[\downarrow]$  keys. **Note:** Take care not to confuse the  $[\uparrow]$  and  $[\downarrow]$  keys with the CURRENT RANGE  $[\uparrow]$  and  $[\downarrow\downarrow]$  keys.

Note that the Model 273A 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 Rate/250, or 250  $\mu$ V, whichever is larger. **Note:** Under remote control via the rear panel GPIB or RS-232C port, steps as small as 25  $\mu$ V (MR = 1), or 2.5  $\mu$ V (MR = 0), can be attained as explained in the *Model 273A Remote-Programming Command Handbook*).

**Example:** Given a scan rate of 1 V/s, the step size will be 4 mV, i.e. (1000/250 = 4)

Each Setup key is discussed in the following paragraphs.

1. [E/I 1, 2 and 3]: These keys are used to specify the potential/current applied at each of three stages in a scan or pulse sequence. Potential is the controlled parameter in Potentiostat mode operation, while current is controlled in Galvanostat operation. [E/I 1] specifies the potential/current applied at the start of the scan. [E/I 2] specifies the potential/current applied at end of the scan's first leg. In Half Cycle mode operation, the scan ends at E/I 2 and E/I 2 continues to be applied. In Single Cycle operation, E/I 2 is a vertex and E/I 3 specifies the final potential, which continues to be applied after the scan is over. In continuous operation, there is repetitive cycling between E/I 2 and E/I 3 until the [STOP] key is pressed. DELAY 1 and DELAY 2 may be used to determine the length of time E/I 1 and E/I 2 are applied (PASS = 4 ms). The Delay at E/I 1 and E/I 2 may be carried out with the cell either on or off. If off, E/I 1 or E/I 2 is applied after the Delay has expired. The delays may also be specified as drift rates in mV/s. See [DELAY I] and [DELAY 2] for details.

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 minus the lowest potential cannot exceed 4 V. Current is specified as a fraction of the selected CURRENT RANGE up to a maximum of two times the current range (one times on the 1 A range).

#### **Examples:**

a. To set and enter an Initial Potential of -0.250 V, key:

[E/I 1] [+/-] [.] [2] [5] [ENTER]

The +/- key reverses the polarity of the expression and can be pressed at any point before [ENTER] is keyed.

b. To set and enter a Vertex Current of 1 mA when operating on the 100 mA range, key:

[E/I 2] [.] [0] [1] [ENTER]

Since the desired current (1 mA) is 1% of the selected Current Range (100 mA), E/I 1 is set to ".01".

[DELAY 1] and [DELAY 2]: These keys are used to specify delay values. There are two
delays. The first, DELAY 1, precedes the scan. The second, DELAY 2, can be interposed
between the first and second legs of the scan. Delays can be specified as a time interval
(seconds) or as a drift rate (mV/s). Delays can also be PASS'ed, in which case the delay is
minimum (4 ms).

If specified as a time interval, the range is .1 s to 100,000 s, and the delay can be done with the cell either ON or OFF. [CELL OFF], a key in the SETUP group, not to be confused with [CELL ON] in the CELL group, determines whether the delay will be performed with the cell ON or OFF. Each time the key is pressed, it toggles between the two setup states, as indicated in the display. If the cell is OFF during the delay, no potential or current is applied. If it is ON, potential or current, as appropriate, is applied.

If the intent is to postpone starting a scan until the open-circuit cell potential settles, the delay is specified in mV/s. Typically, the potential drifts rapidly at first, and then more and more slowly as it asymptotically approaches its final value. The delay will last as long as it has to, measuring the potential all the time, until the drift rate drops below the specified figure. Delay values from 0.001 mV/s to 10 mV/s can be set. Should the drift change polarity, premature termination could occur. Drift-rate delays should always be done with the Cell OFF. It is the user's responsibility to establish the required OFF state with the Scan Setup CELL OFF key.

Note that the cell potential must be monitored for some time to acquire the data needed for the driftrate calculation. Very low settings require long measurement times, up to a maximum of 1000 s.

Once the delay interval ends, the scan begins and the potential (current), if not applied during the delay, is applied. The Cell is ON during a scan, whatever its status during the preceding delay.

#### **Examples:**

a. To set a Cell ON Delay 1 of 15 s, key:

[DELAY I] [1] [5] [SEC] [ENTER]

b. To set a Cell OFF Delay 2 of 0.1 s, key:

[DELAY 2] [0] [.] [1] [SEC] [CELL OFF] [ENTER]

c. To set a Delay 1 of .02 mV/sec, key:

[DELAY 1] [.] [0] [2] [mV / SEC] [ENTER]

d. To bypass Delay 1 in the scan sequence, key:

#### [DELAY1] [PASS] [ENTER]

3. [SCAN 1] and [SCAN 2]: These keys set the scan rate for the two scan legs. [SCAN 1] applies to the scan from E/I 1 to E/I 2. [SCAN 2] applies to the scan from E/I 2 to E/I 3.

Scan rates from 1  $\mu$ V/s to 1 V/s can be specified in the Potentiostat mode (keyed units are mV/s). In the Galvanostat mode, the range is 1  $\mu$ X/s to X/s, where X is the selected current range. [STEP] can also be selected, in which case the applied Potential/Current simply steps as quickly as possible from one level to the other. [STEP] is useful for applying pulse waveforms.

#### **Example:**

a. To set a Scan 1 rate of 1 mV/s, key:

b. To key a Scan 2 rate of 50  $\mu$ A/s with a Current Range setting of 100  $\mu$ A, key:

Since the desired scan rate units are half the Current Range, the keyed value is simply ".5".

c. To substitute a potential step for Scan 1, key:

4. [EXP]: This key is used to set parameters using engineering notation. For example, to set Delay 1 to 1000 seconds, one could key.

[DELAY 1] [1] [0] [0] [0] [ENTER]

or

[DELAY 1] [1] [EXP] [3] [ENTER]

or

[DELAY 1] [1] [0] [EXP] [2] [ENTER]

and so forth. [EXP] can be regarded as belonging to the numeric keypad subset.

5. [FUNCTION]: This key accesses the Setup Functions. Each function initiates an action not available via the other keys. A function is accessed by keying [FUNCTION] followed by the number corresponding to the desired function.

A number of special functions are provided. Although memory space for 90 functions has been provided (range is 10 to 99), the number actually available is much smaller. The first available function is FUNCTION 10, the next is FUNCTION 11, and so forth, up to the highest one defined. A brief description of each follows.

FUNCTION 10, REINITALIZE SYSTEM: This function allows all of the parameter selections to be cleared and the default values to be restored. When it is accessed, the message REINIT SYSTEM? is displayed. At that point the operator presses the YES key or the NO key (1 or 0 or the keypad), as appropriate, followed by ENTER. If YES, all parameter selections are cleared

and default values restored. Note that the effect is *NOT* the same as that which occurs when the power is cycled. When the Model 273A powers down, all parameter values and selections are retained. When it is powered up, most of the values in effect at the time that power shutdown occurred are restored. The exceptions are the errors and SRQ, which are cleared by cycling the power. (See Command Handbook for more information.)

FUNCTION 11, PRINT OUT INSTALLED OPTION: This function allows the user to determine at a glance whether an option is installed in the unit. When accessed, the number corresponding to the installed option is displayed. If a unit is equipped with the 273A/92 option, the number "92" would be displayed.

FUNCTION 12, ENTER LOG REFERENCE: This function allows the user to specify the Current Range for which a full-scale current will yield 0 V out in the LOG mode. When accessed, the message ENTER LOG REFERENCE is displayed. At that point the user keys a number from "0" (1 A range) to "-7" (100 nA range). The number keyed is the exponent if the current range were expressed in engineering notation in units of amperes. The default value is "0" (1 A range).

For example, suppose one wished to specify the 1  $\mu$ A range. The sequence would be:

- 1. Key [FUNCTION] [1] [2] [ENTER]. The message ENTER LOG REFERENCE will be displayed.
- 2. Key [+/-] [6] [ENTER] to specify the 1  $\mu$ A current range.

With this setting, a current of 1  $\mu$ A will yield 0 V at the OUTPUT connector in LOG mode operation.

FUNCTION 13, ENTER INTERRUPT: This function is used to specify the frequency with which current interrupts are performed if operating with Current Interrupt IR Compensation with the instrument under front-panel control. The interrupt period is specified in units of seconds from .004 s to 30 s with the default corresponding to 1 s.

**Example:** To set a current interrupt period of one second, proceed as follows.

- 1. Key [FUNCTION] [1] [3] [ENTER]. The message ENTER IRUPT will be displayed.
- 2. Key [1] [ENTER] to specify an interrupt interval of one second.

FUNCTION 14, OUTPUT TIME CONSTANT: This function provides a digital, low-pass, 6 dB/octave filter at the front-panel OUTPUT connector. The time-constant range for the filter extends from 0 to 30 seconds. The default value is "0."

FUNCTION 15, PEN DELAY: This function allows the user to control the pen-drop delay. When a scan is initiated, the pen has to move to the starting coordinates. Should the pen drop during this homing operation, the record will be marred. Function 15 determines when the pen will drop. The range is 0 to 10 seconds and the default value is 0.5 seconds. The amount of delay required will depend on the recorder.

FUNCTION 16, AUTO LIMIT: This function sets the most sensitive current range to which the Model 273A can automatically range (auto-ranging active). The range is "0" (1 A) to -7 (100 nA). The default value is "-6."

FUNCTION 17, CURRENT INTERRUPT PERCENT CORRECTION: This function sets the percent of correction applied in Current Interrupt IR Compensation. The range is 0% to 200%. The default value is 100%.

*FUNCTION 18, FIRST EXTRAPOLATION POINT:* This function sets the first of the two times used to do the correction extrapolation in Current Interrupt IR Compensation. The range is 2 to 1997. The default value is 10  $\mu$ s on the 1 A range and on the 100 mA range. It is 75  $\mu$ s on all other ranges. **Note:** The sum of the values of Functions 18 and 19 must be  $\leq$ 1999  $\mu$ s.

FUNCTION 19, SECOND EXTRAPOLATION POINT: This function sets the increment from the first time to do the correction extrapolation in Current Interrupt IR Compensation. The range is 2 to 1997. The default value is 10  $\mu$ s on the 1 A range and on the 100 mA range. It is 75  $\mu$ s on all other ranges. **Note:** The sum of the values of Functions 18 and 19 must be  $\leq$ 1999  $\mu$ s.

FUNCTION 20, IOFFSET VALUE PRESENT: This function reads the IOFFSET value present (unlike the SET I OFFSET button on the front panel, which resets IOFFSET to the present reading).

FUNCTION 21, CALIBRATE 273A: This function is used to calibrate the Model 273A.

6. [OVERRIDE]: This key allows the user to make specific modifications to the setup routines. An override is accessed by keying [OVERRIDE] followed by the number corresponding to the Override wanted. At that point the override's name and status are displayed. The [1 YES] and [2 NO] keys are then used to specify the status of the selected override. The state can be reversed with the YES or NO key, as appropriate. [1 YES] and [2 NO] are dual function keys. YES and NO apply to the overrides; "1" and "2" belong to the numeric keypad.)

A number of overrides have been provided to extend the Model 273A's flexibility, power, and ease of operation. Although memory space for 90 Overrides has been provided (range is 10 to 99), the number actually available is much smaller. The first available Override is OVERRIDE 10, the next is OVERRIDE 11, and so forth, up to the highest one defined. Once one of the overrides has been called up, the Scan Setup UP/DOWN ARROW KEYS can be used to reach the others via scrolling. A brief description of each follows.

OVERRIDE 10, OFFSET DISPLAY I: This Override determines whether I Offset, as set by the SET I OFFSET key in the OUTPUT control group, will influence the displayed current. If YES, I OFFSET will affect both the level at the OUTPUT connector and the displayed current. If NO (default status), only the level at the OUTPUT connector will be affected.

OVERRIDE 11, UPDATE COULOMBS: This override determines whether the coulombs indication will be periodically updated as the experiment progresses. If YES (default status), the displayed coulomb accumulation will be updated every timebase. If NO, coulombs are not measured. NO is normally selected if speed of data acquisition is the main concern, such as might be the case in remote control experiments.

If the Model 273A is being controlled from an external computer via the RS-232C or GPIB interface, the rate at which data can be taken will be faster with this override in the NO state.

OVERRIDE 12, CORRECT DISPLAYED E: This override determines whether the displayed potential will be affected by the IR compensation. If YES (default state), the displayed potential will be corrected for IR drop (assuming that IR Compensation is in use), the same as the potential at the E MONITOR and recorder outputs. If NO, any errors due to IR drop will affect the displayed potential as well, even though IR Compensation is in use. **Note:** IR drop errors always affect the rear-panel ELECTROMETER MONITOR output, whether or not IR Compensation is active.

OVERRIDE 13, CELL OFF AT END: This override determines the Cell ON/OFF status at the end of a scan. If YES, the cell will be turned off (counter electrode connection interrupted) at the end of the scan. If NO, the counter electrode connection remains intact and the final E/I continues to be applied. Note that in the CONTINUOUS control mode, the experiment doesn't end until the STOP key is pressed. At the point, the cell status is that determined by this override.

OVERRIDE 14, LINE SYNC: This override determines whether data acquisition will be synchronized with the power line frequency. If YES, samples are taken every 16.6 ms (60 hz power) or every 20 ms (50 Hz power). If NO, samples are taken at the rate determined by the internal time base. The default state of this override is NO.

Note that, if the Override 14 status is changed while a scan is in progress, the scan will stop. It can be restarted only by pressing the START key.

- 7. [1 YES] and [2 NO]: As previously explained, these are dual-function keys. In specifying an Override, they act as YES/NO designators. In keying a parameter value, they simply specify the numbers "1" and "2." In this capacity, these keys are part of the numeric keypad.
- 8. NUMERIC KEYPAD: This subset of the Scan Setup keys consists of [1 YES], [2 No], [3], [4], [5], [6], [7], [8], [9], [0], [.], [+/-], and [EXP]. These keys are used to actually key the parameter values. As each key is pressed, the corresponding character appears in the display. As explained in the previous two paragraphs, [1 YES], and [2 NO] have alternate functions.
  - [EXP] precedes an exponent to the base 10 and is used to express values in engineering notation (see "6" for examples).
  - [.] simply sets the decimal point. As many as three digits can follow it.
- 9. [+/-] sets the polarity. As an expression is keyed, it has a positive value. The first time that [+/-] is pressed, independent of the parameter's previous polarity, it sets the parameter negative. Each subsequent time the key is pressed, the polarity reverses.
  - Note that [+/-] can be pressed at any time prior to pressing [ENTER]. However, in an expression containing EXP, once [EXP] is keyed, the [+/-] sign will only affect the polarity of the exponent.
- 10. [↑] and [↓]: These keys have a three-fold function. First, during the setup process, they can be used to make small adjustments in the value of the displayed parameter. Each time an arrow key is pressed, an incremental change occurs (1 mV for potential, 0.1% for current). If the key is held in, the parameter value increments or decrements continually. The rate of change increases by a factor of ten if the key is held in for several seconds. It is *not* necessary to press the ENTER key to execute the change.

Second, they can also be used to increment or decrement a parameter during an actual scan. The active parameter is the one operated on.

Third, they can be used to scroll through the various Override messages. The user can bring one Override message to the screen, and then use the arrow keys to scroll through the available choices to reach the one of interest.

11. [STEP]: This key is used to substitute a step between two levels instead of a scan. The applied potential/current will step as quickly as possible between the two levels. Another operation cannot be initiated for 4 ms. The step feature allows the Model 273A to perform double-potential step chronoamperometry, in which the applied potential starts at E/I 1, remains there long enough to let the sample come to equilibrium with the solution, steps to E/I 2 for DELAY 2 seconds, and then steps to E/I 3, which continues to be applied.

**Example:** To substitute a step for SCAN 1, key:

## [SCAN 1] [STEP] [ENTER]

12. [PASS]: This key allows one or both delays to be dropped from the scan sequence. A passed delay is 4 ms.

**Example:** To drop DELAY 2 from the scan sequence, key:

[DELAY 2] [PASS] [ENTER]

13. [SEC]: This key is used in conjunction with either of the DELAY keys to specify a delay interval in seconds.

Example: To set a DELAY 1 interval of 100 s, key:

[DELAY 1] [1] [0] [0] [SEC] [ENTER]

14. [mV/SEC]: This key is used in conjunction with either of the DELAY keys to specify a delay interval in drift units. Defining the delay in this manner is particularly well suited to experiments where the specimen must be allowed to come to equilibrium before valid measurements can be taken. The open-cell potential is monitored, allowing the drift rate to be measured as it asymptotically approaches the rest potential. When the actual rate becomes slower than the rate specified via the [mV/SEC] key, the delay interval ends. The slower the specified drift rate, the longer the minimum delay time (the longer it takes to get a valid drift-rate reading).

**Example:** To set a DELAY 2 drift rate of 0.01 mV/s, key:

[DELAY 2] [.] [0] [1] [mV/SEC] [ENTER]

15. [CELL OFF]: This key determines whether delay intervals specified in seconds will be done open-cell or with the specified potential/current applied. The key is a toggle, that is, its state (indicated by the alphanumeric display) reverses each time the key is pressed.

**Example:** To set an open-cell DELAY 1 of 100 s, key:

[DELAY 1] [1] [0] [0] [SEC] [CELL OFF] [ENTER]

16. [ENTER]: This key has two functions. First, it enters newly specified parameter values into the setup program. Second, when not entering a parameter, it allows different kinds of information to be scrolled through the bottom line of the display.

For example, when a setup parameter key is pressed, the set value is displayed, together with the message, ENTER XXX (XXX signifies the parameter). The new value can then be keyed and ENTER pressed, after which the new value will be displayed. Pressing ENTER again clears the new value from the display and establishes the scroll mode, in which the bottom display line scrolls through several possible display options, advancing once each time the ENTER key is pressed. The choices are:

- a. Second Display Line Blank.
- b. Charge in Coulombs.
- c. Log of I if LOG is Enabled.
- d. Status of the Scan. Possibilities include:
  - i. Delay Time Remaining (Delay specified in seconds).
  - ii. Time Remaining Until Drift Measurement Can be Made (Delay specified in mV/s). Alternatively, measured drift rate if measurement has taken place.
  - iii. Stopped Scan.

iv. E Applied or I Applied, according to selected mode.

**Note:** If the scan proceeds too fast for the bottom line of the display to be updated in real time, the message "SCANNING" will be displayed on the bottom line.

## 5.2.5. INTERFACE Group

This area of the panel contains five indicators and a pushbutton. They provide useful information when controlling the Model 273A from a remote computer. When the instrument is under front-panel control, all five indicators should be extinguished and the LOCAL key will have no function.

When controlling from a remote computer via the GPIB or RS-232C port, all five indicators and the pushbutton are active, although SRQ, REMOTE, and the pushbutton apply to GPIB communications only. A brief description of each follows.

- TALK: This indicator lights when the Model 273A has output ready to send to the host computer. It goes out when the Model 273A has finished sending this output. The TALK indicator does NOT signal that the host computer has transmitted a TALK message. Rather it indicates that the Model 273A has more output to dump before the command is completed.
- 2. LISTEN: This indicator lights when the Model 273A senses the first character of a command. It remains lighted until the terminator is sensed. The LISTEN indicator does NOT signal that the host computer has transmitted a LISTEN message. Rather it indicates that the Model 273A is expecting more input before the current command is fully defined.
- 3. REMOTE: This indicator lights when the Model 273A is under remote control via the GPIB interface, that is,  $\overline{REN}$  is asserted and the Model 273A's LISTEN address has been applied. As long as the REMOTE indicator is lighted, the Model 273A's front panel is "locked out." Return from remote to local operation can be accomplished simply by pressing the adjacent LOCAL key (only effective if the LOCAL LOCKOUT message hasn't been applied). Other ways of returning to local include applying the GO TO LOCAL message, deasserting  $\overline{REN}$ , or cycling the power. **Note:** LOCAL LOCKOUT and GO TO LOCAL are GPIB messages defined by IEEE-488.
- 4. SRQ: This indicator lights when \$\overline{SRQ}\$ is asserted, that is, when the Model 273A has initiated a service request. (The use of SRQ and serial polling to control GPIB communications is discussed in Subsection B.4 of Appendix B of the *Model 273A Remote-Programming Command Handbook.*) It remains lighted until the controlling computer completes a serial poll of the Model 273A or the power is cycled.
- 5. ERROR: This indicator lights if there was an error in the most recently executed command. The nature of the error can be determined by evaluating the ERR command response.
- 6. (LOCAL): This key returns the Model 273A from GPIB control to front-panel control (See Appendices A and B of the *Model 273A Remote-Programming Command Handbook* for a discussion of GPIB Communications Considerations.) It is effective only if the GPIB LOCAL LOCKOUT message has not been applied. Also, as long as REN continues to be asserted, applying the Model 273A's LISTEN address will immediately restore GPIB control. Note that this key does not affect control of the Model 273A via the RS-232C Interface, which is always active.

If the LOCAL key is pressed while executing a multiple remote command, an error will occur because there will be a remote/local violation. The host computer can avoid this possibility by sending LOCAL LOCKOUT (a GPIB message), which disables the effect of the LOCAL key.

#### 5.2.6. CONTROL Group

- 1. SCANNING: This indicator is off at power-up and on during each active scan. At powerup, the E/I APPLIED indicator is lighted, indicating that the 273A is ready to apply the previously speed E/I APPLIED level. (It won't actually be applied, however, unless both the CELL switch and the CELL ENABLE switch are ON.) If a scan is initiated by pressing START, the E/I APPLIED indicator goes out and the SCANNING indicator lights, signifying that a scan is underway. The progress of the scan can be monitored by observing the Scan Status Display. When the scan ends, either by pressing STOP or by completing the programmed sequence, the SCANNING indicator will go out. One of the three Scan Status Display Indicators, E/I 1, E/I 2, or E/I 3, will remain lighted. An Override (see Section 5.2.4) determines whether the cell will be ON or OFF.
- 2. HOLDING: This indicator lights if the HOLD/CONTINUE key has been pressed during a scan. Pressing the HOLD/CONTINUE key causes the scan to halt until the key is pressed again (CONTINUE), at which point it resumes and the HOLDING indicator extinguishes.

If a HOLD occurs when a potential/current is applied, it will continue to be applied, subject to the usual Cell switch and Cell Enable switch constraints. This is true if the hold takes place at any time during the scan sequence (E/I 1, DELAY 1, SCAN 1, E/I 2, DELAY 2, SCAN 2, E/I 3). If the hold occurs during a delay measured in seconds, time is not counted for the duration of the hold. If it occurs during a delay measured in drift, the drift rate is not measured during the hold. If applied during SCAN 1 or SCAN 2, the scan simply halts and the potential/current in effect as of the moment of the halt continues to be applied.

- 3. [START]: This key is used to start a scan. When it is pressed, E/I 1 lights and the scan progresses as programmed.
- 4. [HOLD/CONTINUE]: This key is used to start a hold interval as described in the discussion of the HOLDING indicator. Pressing the key a second time continues the measurement sequence.
- 5. [ADVANCE]: This key's function differs according to whether a scan sequence is in progress when it is pressed.

If pressed during a scan, it causes the sequence to immediately advance to the next step. For example, if pressed during the DELAY 1 interval, the sequence would immediately advance to SCAN 1. Similarly, 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.

If pressed at any other time, the message "NOT SCANNING" is displayed.

- 6. [STOP]: This key stops a scan sequence, immediately establishing the end-of-scan condition, and E/I 1 on the Scan Status Display lights. The applicable Override (see Section 5.2.4) determines whether the cell will be ON or OFF.
- 7. CYCLE CONTROL: The Cycle Control key allows selection of three different cycle modes: HALF CYCLE, SINGLE CYCLE, and CONTINUOUS. A separate indicator is provided for each and one of the three will always be lighted. Selection is accomplished with the associated key. Each time the key is pressed, the selection increments, the sequence being HALF CYCLE to SINGLE CYCLE to CONTINUOUS and from there back to HALF CYCLE. A brief description of each follows.
  - a. HALF CYCLE: This is the simplest cycle, involving only two potential/current settings. A half-cycle sequence proceeds as follows.
    - i. DELAY 1 at E/I 1.

- ii. SCAN1 from E/I 1 to E/I 2.
- iii. Scan STOPS on reaching E/I 2.

During DELAY 1 the cell can be either ON or OFF, as programmed. During SCAN 1 the Cell will be ON. At the end of the scan, the E/I 2 indicator will remain lighted. The cell will remain ON or OFF according to the state of the applicable Override.

- b. SINGLE CYCLE: A Single Cycle is more complex and involves all three programmed potential/current levels. The sequence is:
  - i. DELAY 1 at E/I 1.
  - ii. SCAN1 from E/I 1 to E/I 2.
  - iii. DELAY 2 at E/I 2.
  - iv. SCAN 2 from E/I 2 to E/I 3.

Scan STOPS on reaching E/I 3.

During DELAY 1 and DELAY 2 the cell can be either ON or OFF, as programmed. During SCAN 1 and SCAN 2 the Cell will be ON. At the end of the scan, the E/I 3 indicator will remain lighted. The cell will remain ON or OFF according to the state of the applicable Override.

- CONTINUOUS: Continuous is the most complex of the three cycles. The sequence is as follows:
  - i. DELAY 1 at E/I 1.
  - ii. SCAN 1 from E/I 1 to E/I 2.
  - iii. DELAY 2 at E/I 2.
  - iv. SCAN 2 from E/I 2 to E/I 3.
  - v. DELAY 1 at E/I 3.
  - vi. SCAN 1 from E/I 3 to E/I 2.
  - vii. Steps iii through vi repeat until STOP key is pressed.

During DELAY 1 and DELAY 2 the cell will be either ON or OFF, as programmed. During SCAN 1 and SCAN 2 the cell will be ON. Automatic cycling of the scan, back and forth from E/I 2 to E/I 3, will continue until halted by pressing the STOP key. (The cycle returns to E/I 1 when STOP is pressed.) The cell will be ON or OFF according to the state of the governing Override.

8. [E/I APPLIED]: This key allows a speed constant potential/current to be applied to the cell. If the associated indicator is lit, it means that E/I APPLIED will be applied if the Cell is ON. The E/I Applied indicator is lighted at powerup. It extinguishes when the START key is pressed to begin a scan. The value for the E/I APPLIED parameter is that in effect at the time the instrument was last powered down, or the last value set if changed since powerup. E/I APPLIED can take potential values from -10 V to + 10 V. Current values are a fraction of the selected Current Range up to the maximum possible value of "2" ("1" is maximum on the 1 A range).

There are two ways of changing the value of E/I APPLIED. The simplest is to use the [1] and [1] keys. Anytime a potential/current is applied, these keys can be used to increment

(decrement) the potential in 1 mV steps or the current in 0.1% of Current Range steps. If one of these keys is held down continuously, the steps increase to 10 mV or 1% of Current Range, allowing a large change to be achieved quickly. The second way of changing the parameter's value is to specify a specific value and enter it.

Example: To set E/I APPLIED to 1 V, key:

[E/I APPLIED] [1] [ENTER]

#### 5.2.7. MODE Group

STABILITY versus SPEED: This subgrouping consists of two indicators and one key. One of
the two available modes, HIGH SPEED or HIGH STABILITY, will always be in effect and the
corresponding indicator will glow. The key is used to alternate between the two modes.
Stability/Speed selection is entirely independent of whether the instrument is operated as a
potentiostat or galvanostat.

The HIGH SPEED mode gives the fastest response (see specifications) in Potentiostat or Galvanostat operation, but without guaranteed stability. 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 HIGH STABILITY mode provides extreme stability at somewhat reduced speed (see specifications). 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 HIGH STABILITY mode, it usually results from a high-resistance reference electrode. Even a well-designed, low-impedance reference electrode can develop a high resistance if salts precipitate in the junction.

- OPERATING MODE: There are three basic operating modes, each with its own key and indicator. One of the three modes will be in effect at all times as indicated by the associated indicator. To transfer from the mode in effect to either of the other two modes, simply press the target-mode key.
  - a. POTENTIOSTAT: In this mode the 273A controls the potential of the working electrode with respect to the reference electrode. The potential at the counter electrode is driven to the potential required (consistent with the ±100 V compliance of the Control Amplifier) to establish the desired working electrode potential.
  - b. GALVANOSTAT: In this mode the 273A controls the cell current at the specified faction of the selected Current Range (up to the maximum of two times the Current Range; one times the range if 1 A is selected). The counter electrode is driven to the potential required (consistent with the ±100 V compliance of the Control Amplifier) to establish the desired cell current. No reference electrode is required, although one can be used to monitor the potential, if desired.
  - c. MEASURE ONLY: The open-circuit cell potential is continuously monitored and displayed in this mode. Neither the potential nor the current is controlled. The cell is OFF, even if Cell ON has been previously selected with the Cell key. If an attempt is made to turn the Cell ON while in the Measure Only mode, the message: CAN'T TURN CELL ON IN MEASURE ONLY is displayed. See 5.2.9 for a discussion of the effect of a voltage applied to the External Input on the signals routed to the electrochemical cell.

## 5.2.8. CELL Group

There are two keys and one indicator in this group. All three are associated with the internal switches that provide the connection to the counter electrode (red clip lead). The CELL ENABLE pushbutton has two positions, in (ON) and out (OFF). CELL ENABLE is a hard-wired, fail-safe

switch. Whenever it is in the OFF position, there is no possible way, including commands from an external computer, for voltage to be applied to the counter electrode. This switch is provided to assure operator safety when working on the cell.

#### Warning!

When working with the cell, including making and breaking connections, the CELL ENABLE switch must be in the off position. The ±100 V output and high current capability of the 273A make it a dangerous device, one capable of causing severe injury or even death. The connections should be handled only

The upper Cell key provides the normal Cell ON/OFF control during operation. Unlike the CELL ENABLE switch, this key is subject to software override. For example, the ON state must be selected before starting a scan. However, a scan may contain delay intervals to be carried out in the Cell OFF state. If this is the case, the cell is turned off automatically at the appropriate points in the measurement sequence.

The Cell indicator light gives the cell status at all times. This red indicator has three states, OFF, HALF BRIGHT, and FULL BRIGHT. The significance of each state is:

**OFF:** Both the CELL key and the CELL ENABLE switch are OFF, or the CELL key is OFF and the CELL ENABLE switch is ON.

HALF BRIGHT: CELL key is ON but the CELL ENABLE switch is OFF.

**FULL BRIGHT:** Both the CELL key and the CELL ENABLE switch are ON. The cell is ON only when the light is at full brightness.

The Model 273A is controlled even when the cell isn't ON, that is, having the Cell OFF does not cause an overload condition.

In the context of this discussion, CELL ON and CELL OFF refer to the path from the Control Amplifier output to the Counter Electrode cable lead. The red indicator does not respond to the status of the actual counterelectrode connection. That connection could be secure or faulty and it would have no effect on the red indicator, which responds only to the CELL ON switch, the CELL ENABLE switch, and on the governing software. However, if the Cell is ON and there is no connection to the counter electrode, the effect will be the same as if the cell had infinite resistance and an E OVERLOAD indication will occur.

#### 5.2.9. INPUT Group

This group contains one indicator, one key, and one BNC connector. Together they provide the means for summing external control potentials with those generated in the instrument or transmitted to it from an external computer via the rear-panel RS-232C or GPIB port. The external control potential is applied to the EXTERNAL INPUT connector. In potentiostat operation, voltages from -10 V to + 10 V can be applied. In galvanostat operation, the range is -2 V to +2 V (corresponding to  $\pm 2$  times the full-scale current), except on the 1 A Current Range where the range is restricted to  $\pm$  1 V. The input impedance is 100 k $\Omega$ .

In potentiostatic operation, all of the control potentials add algebraically. For example, if the voltage applied to the EXTERNAL INPUT is +0.5 V, at a time when a programmed potential of +0.5 V is applied, the net potential seen by the Model 273A will be + 1.0 V, and the working electrode would be controlled at + 1.0 V with respect to the reference electrode. 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 current more positive by any means *except* applying a potential to the EXTERNAL INPUT connector will tend to make the current more cathodic; making the applied current more negative will tend to make the current more anodic. This sense is reversed at the external 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 the EXTERNAL INPUT is a high-impedance potential input in both potentiostatic and galvanostatic operation. In potentiostatic operation, a volt at the External Input results in a volt at the working electrode (other control sources zero). In galvanostatic operation, a volt at the External Input results in full-scale cell current (other control sources zero).

The EXTERNAL INPUT is controlled by the EXT ON key. When pressed, the associated indicator lights and the Model 273A becomes responsive to potentials applied to the EXT INPUT connector. If pressed again, the indicator goes out, indicating that potentials applied to this input will be ignored.

## 5.2.10. FILTER Group

This group contains two indicators and a key. The key allows the user to select OFF (no filter) or LOW PASS (low-pass filter selected). The indicator corresponding to the filter status will always be lighted.

The low-pass filter has a sharp cutoff at 5.3 Hz. (A 590 Hz low-pass filter is also available under software control from the back panel.) It is often helpful when making low-current measurements on large electrodes because it reduces the noise at the OUTPUT connector. The disadvantage of using the filter is that the Output response time is degraded. The filter has no effect on I MONITOR output. Use of the filter for galvanostatic operation is not recommended.

It may be advantageous to synchronize data acquisition with the power line frequency when filtering. This can be done using Override 14, if desired. The use of Override 14 is described in Section 5.2.4.

#### 5.2.11. IR COMPENSATION Group

#### Introduction

There are three keys and two indicators in this group. Positive-Feedback IR Compensation is selected by pressing the POSITIVE FEEDBACK key. Current Interrupt IR Compensation is selected by pressing the CURRENT INTERRUPT key. Both keys are toggles for the corresponding function. However, 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 drop out and the newly selected mode will become active. The associated indicators indicate the status of the IR Compensation modes. The SET IR key is used in establishing Positive Feedback IR Compensation. Note: Positive feedback is not available when the Model 273A is operating in the galvanostatic mode.

The voltage at the front-panel E MONITOR connector and at the rear-panel RECORDER INTERFACE connector is corrected when either technique is employed, in contrast to the voltage output at the rear-panel ELECTROMETER MONITOR connector, which is not corrected for the iR drop.

The two IR Compensation techniques are performed in entirely different ways. Because the techniques each have strengths and weaknesses, some situations will call for one technique and some for the other. Table 2 briefly lists the relative strengths of the two techniques. Both techniques are discussed in the following paragraphs.

Table 2. POSITIVE FEEDBACK AND INTERRUPT COMPARISON

PARAMETER INTERRUPT	POS. FEEDBACK	CURRENT
SPEED	FAST	SLOW
EASE OF USE	MORE DIFFICULT	VERY SIMPLE
R <sub>u</sub> TRACKING	DOES NOT TRACK	TRACKS
STABILITY	CRITICAL	VERY STABLE
ARTIFACTS	NONE	MINOR
E MON. CORRECTED	YES	YES
GALVANOSTAT MODE	NOT AVAILABLE	AVAILABLE

#### Positive Feedback IR Compensation

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 exactly the value required to compensate for  $R_{\rm u}$ . 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 273A. While the 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.
- 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<sub>u</sub> is frequently not stable; it can change with the applied potential, time, and changes in cell geometry, etc.

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.

Resistance values have a resolution limited to 0.1% of 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 this feature with autoranging. The resistance resolution limit and maximum compensation as a function of Current Range is:

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

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

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  $\Omega$ , the actual programmed resistance will change to 1234  $\Omega$ . 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  $\mu$ A, where the resolution limit is 100  $\Omega$ , the actual programmed resistance will become 1200  $\Omega$  because the value can only be represented to the nearest 100  $\Omega$ . In other words, the error will now total 34  $\Omega$ . Similarly, a further shift to 1  $\mu$ A would give a resistance of 1000  $\Omega$  (234  $\Omega$  error) and a shift to 100 nA would give a resistance of 1000  $\Omega$  (1234  $\Omega$  error). **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 key 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.

#### 1. R<sub>u</sub> KNOWN:

- a. Press "SET IR." The current value of assumed R<sub>11</sub> will be displayed.
- b. Key the value of  $R_{\scriptscriptstyle u}$  to be compensated and press ENTER. The new value will be displayed.
- c. Press "POSITIVE FEEDBACK." The associated indicator will light and positive feedback compensation will be established. **Note:** This pushbutton is a toggle; to turn the compensation off, simply press POSITIVE FEEDBACK again.

## 2. R<sub>u</sub> NOT KNOWN:

- a. Press SET IR. Then key and enter an R, of "0." 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.
- b. Press the POSITIVE FEEDBACK key, establishing Positive Feedback mode operation.
- c. Program the Model 273A to apply a 62.5 Hz, ±50 mV square wave to the cell. The following parameter values can be used. **Note:** The ±50 mV figure is not critical. Smaller or larger amplitudes, if more appropriate to the chemistry, can be used.

**Note:** If LINE SYNC is ON (OVERRIDE 14), 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 off (less flicker on the oscilloscope display).

E / I 1 -0.05 V with respect to the potential of interest

DELAY 1: PASS (see Note)

SCAN 1 STEP

E / I 2 +0.05 V with respect to the potential of interest

DELAY 2 PASS (see Note)

SCAN 2 STEP

E / I 3 -0.05 V with respect to the potential of interest

Control Mode CONTINUOUS

**Note:** 62.5 Hz rate results from setting both delays to PASS.

- d. Establish the Cell ON state (CELL ON and CELL ENABLE). Then press [START] to apply the square wave to the cell. The cell should be completely set up for the measurements to be made. (Any change at the cell will probably change R<sub>II</sub>.)
- e. Monitor the I MONITOR connector with an oscilloscope. The Model 273A should be set to the same current range as will be used in the forthcoming measurement. If the Current Range setting is changed, the actual value of IR compensation can change as previously discussed. At the oscilloscope, use internal triggering and display the waveform so that the waveform's risetime can be readily observed. A horizontal deflection factor of 2 ms per division will generally give good results.
- f. Press SET IR so that the set value of  $R_U$  (it should be "0") is displayed on the *Model 273A*.
- g. While observing the displayed waveform on the oscilloscope, hold in the Scan Setup [†] key. The R<sub>u</sub> compensation will increase, causing the decay intervals in the displayed waveform to become shorter. The longer the key is held in, the greater will be the applied compensation. The actual value can be directly read from the Model 273A display.

Figure 6 illustrates the compensation's effect. 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  $R_{\upsilon}$ ) could cause the potentiostat to oscillate. Compensation in the 95% to 100% range is best suited to slow experiments (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.

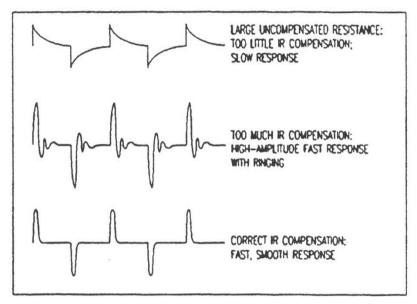


Fig. 6. I Monitor Waveforms as a Function of  $R_{\text{U}}$  Compensation (Square Wave Applied).

When you are satisfied with the compensation level, turn off the cell and do the Scan Setup appropriate to the intended measurement. Unless changed, the set compensation level will remain in memory, ready to be applied whenever POSITIVE FEEDBACK is activated.

#### **Current Interrupt IR Compensation**

In this technique the current is periodically interrupted and a correction function developed that is based on the time versus potential change that occurs at the moment of interrupt. Specifically, the cell is disconnected (in less than 1  $\mu$ s) for  $\approx$  190  $\mu$ s at periodic intervals. (As fast as the interrupts are, they nevertheless do affect the experiment, and a small artifact may be noticed in the output for each interrupt.) At disconnect, a drop in the Electrometer potential equal to iR<sub>U</sub>, occurs.

Thirty-two potential points are then taken at 5  $\mu$ s intervals. Two of these points are used to define a projection that is extrapolated back to the instant just after current interruption. By comparing the potential at that instant with that before the interruption, a correction function is developed that corrects for the uncompensated IR drop in the cell.

Several functions (see Section 5.2.4) are involved in setting the Current Interrupt parameters. FUNCTION 13 sets the interval between current interrupts. The range is .004 s to 30 s, with a default setting of 1 s. (In doing Curve Acquisition with the 273A controlled from a host computer, the interrupt timing is controlled by the computer.) FUNCTION 17 sets the percentage of correction, adjustable from 0% to 200%. FUNCTION 18 selects the first of the two extrapolation points. The range is 2 to 1997  $\mu$ s. The default selection is 10  $\mu$ s on the 1 A and 100 mA Current Ranges, and 75  $\mu$ s on all other Current Ranges FUNCTION 19 selects the time increment used to determine the second point. The selection range for the increment is also 2 to 1997  $\mu$ s. The default selection is 10  $\mu$ s on the 1 A and 100 mA Current Ranges and 75  $\mu$ s on all other Current Ranges.

**Note:** The default values give good results in many applications. Do not change these values using the applicable functions unless you are well grounded in current-interrupt IR compensation theory. This is particularly true of functions 17, 18, and 19. A prerequisite to evaluating these parameter selections is that an oscilloscope be connected to the rear-panel electrometer monitor connector to observe the current-interrupt waveform, as discussed in the following paragraphs. Even using 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 7 illustrates the rear-panel ELECTROMETER MONITOR 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 routine is to measure this drop so that  $R_U$  can be calculated and a correction factor developed.

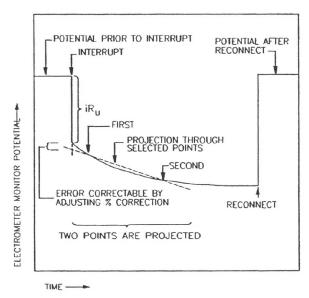


Fig. 7. Current Interrupt Waveform at Electrometer Monitor Connector.

After interrupt, thirty-two samples are taken at  $5 \mu s$  intervals. Two of these are selected (Functions 18 and 19) and extrapolated back to the moment of interrupt. The goal is to select two points that will provide a projection that exactly 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.

Fortunately, Function 17, 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 Function 17 to set the % Correction to 200%. Similarly, if the intersection occurred below the drop, as in the depicted example, Function 17 would be used to set a % Correction of less than 100% (90% would be about right for the example). Once the parameters are set via the required functions and Current Interrupt IR Compensation established, no other operator intervention is required during the measurement.

Current Interrupt IR Compensation has several advantages with respect 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 press of a button to enable the correction. (The default values of Functions 13, 17, 18, and 19 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.

The procedure for establishing Current Interrupt IR Compensation is very simple. Simply press the CURRENT INTERRUPT key. The associated indicator will light and the iR drop will be automatically compensated in subsequent measurements. To turn the technique OFF, simply press the CURRENT INTERRUPT key again (the indicator light will extinguish). **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 rear-panel ELECTROMETER MONITOR connector with an oscilloscope with the goal of optimizing the Current Interrupt Parameters as established by Functions 13, 17, 18, and 19. The selection of the extrapolation points and the % Correction setting determine the accuracy of the applied correction. Bear in mind that the waveform width (interval between disconnect and reconnect) is  $\approx 190~\mu s$ . The thirty-two samples (spaced at 5  $\mu s$  intervals) are located, equally spaced, in the first 160  $\mu s$  of the waveform. Note that the waveform depicted in Fig. 7 is idealized; actually encountered waveforms may not be so easy to analyze.

#### 5.2.12. E Monitor

The potential of the working electrode with respect to the reference electrode is continuously provided at this connector. This same signal is provided to the Recorder X Drive signal line at the rear-panel RECORDER INTERFACE connector. A meaningful E MONITOR output is provided as long as there is a reference electrode. If there is no reference electrode, such as would normally be the case in Galvanostatic operation, the E MONITOR output is undefined. In Potentiostatic operation, if IR COMPENSATION is on (either technique), the compensation corrects the E MONITOR output level for the IR drop.

**Note:** This is not the case for the rear-panel ELECTROMETER MONITOR output, which is not compensated.

The E MONITOR signal is analog and quite clean, free of the steps, spikes, and similar interference common to digitally produced signals. The output impedance is 1 k $\Omega$ . It is inverted with respect to the applied potential. For example, if the applied potential is + 1 V, the potential at the E MONITOR connector will be -1 V. Note that the potential at the rear-panel ELECTROMETER MONITOR connector is also inverted with respect to the applied potential.

#### **5.2.13. CURRENT RANGE Group**

There are three keys, nine indicators, and one BNC connector in this group. Their function is to set and monitor the Model 273A's full-scale current range. One of the eight available current ranges is always selected, and the corresponding indicator lit. A cell current equal to the selected current range gives 1 V at the I MONITOR connector. A current of two times the current range generates an I OVERLOAD condition, except on the 1 A range where the limit is 1 A.

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

There are two selection modes, MANUAL and AUTO. In the MANUAL Mode, the range is selected with associated arrow keys. The  $\uparrow \uparrow$  key moves the selection upwards through the available ranges. The  $\downarrow \downarrow$  key moves it downwards.

In the AUTO mode, the Model 273A automatically seeks the current range that causes the I MONITOR Output to be between 15% and 190% of full scale, that is, between 150 mV and 1.9 V. If the current level is less than 15 nA, the 100 nA range will be selected. If it is more than 1 A, the 1 A 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 can be lost if the data amplitude is changing faster than it can be tracked by the auto-ranging function. Auto-ranging is restricted to CONTROL E operation. Should the user attempt to select Auto Ranging in CONTROL I operation, an error message will be generated.

Automatic current ranging is additionally affected by the LOW PASS filter, if selected. If LOW PASS is selected, there is a 150 ms dead time after each range change. No further current ranging can occur during this dead time, which is inserted to allow the input to settle before making a new ranging decision.

Note, however, that an overload, even if it occurs during a dead-time interval, will always force a range shift to the next higher current range, and that the overload detection circuit is not affected by the presence of a filter.

## 5.2.14. OUTPUT Group

There are three keys, three indicators, and a BNC connector in this group. A voltage dependent on the cell current or transferred charge is available at the OUTPUT connector (identically the rear-panel Recorder Y-Axis Output). The other items in the group either set or indicate the nature of the dependency.

- 1. OUTPUT: An analog of the cell current is available at this connector. This output tracks the current (linear or log) or transferred charge (coulombs). The output impedance is 1 k $\Omega$ . In the linear mode, the output range is  $\pm$  2 V ( $\pm$  1 V full scale). In the other modes it is  $\pm$  10 V. Note that the voltage at this connector is derived from a 12 bit digital-to-analog converter that is updated each time the applied potential or current changes. These updates may be marked by the presence of moderate digital switching transients at this output. For best results, use the I MONITOR output to track current unless use of the low-pass filter is desired.
- COULOMBS, LINEAR, LOG: These three lights indicate the output mode as selected by the associated key. There is additionally a fourth mode, OFF, established when the other three indicators are extinguished. The output in each mode is as follows.
  - a. OFF: 0 V is present at the OUTPUT connector.
  - b. LINEAR: The voltage varies linearly with the current. A full-scale current gives 1 V out. The maximum output, as dictated by the maximum current, is 2 V, corresponding to a current of two times the selected current range (1 V maximum on 1 A range). Low currents on a fixed current range may show the stepped response of a digitally reconstructed signal.
  - c. LOG: The voltage varies with the log of the current. A current equal to the Log Reference Range (Function 12, Section 5.2.4) gives 0 V out. The transfer function is 1 V per decade, consistent with the constraint that the output cannot exceed ±10 V and currents larger than two times full scale will cause an overload condition. For example,

on the 1 mA current range (assuming a Log Reference of -3), 1 mA would give 0 V, 100  $\mu$ A would give -1 V, 10  $\mu$ A would give -2 V, and so forth.

Currents larger than the Log Reference Range give positive outputs. In the example being considered, a current of 2 mA would give +0.301 V on the 1 mA range, a current of 10 mA would give 1 V, and a current of 100 mA would give 2 V.

These relationships hold for both automatic and manual current ranging.

The maximum possible voltage in any case is 10 V. The smallest change is 5 mV (1 part in 2000).

d. COULOMBS: In this mode, a full-scale current for one second on the current range in effect when RESET INTEGRAL is pressed will give 10 V out. For example, if, on the 1 mA range, the current was 1 mA for 1 s, the OUTPUT voltage would be 10 V, representing one millicoulomb of charge. At that point, the output scaling would decrease by a factor of ten. In the example, after one second, the output would reach 10 V and then immediately drop to 1 V as the scaling shift occurs. The voltage would then rise linearly with charge until 10 V was reached again (this time indicating ten millicoulombs). At that point, another factor-of-ten scaling change would take place, and the cycle would repeat. This process can repeat as many times as necessary for any practical experiment. The current integral, in coulombs, can be displayed, if desired, with continuous updating as the measurement progresses. (Recall that one of the functions of the [ENTER] key is to control the "bottom line display".)

If anodic to cathodic crossover occurs during the measurement, the coulometer will reverse direction and downrange if necessary. Current Range changes that occur, either manual or automatic, will have no effect on the voltage at the OUTPUT connector in coulometer operation.

For best results, the starting current range, the one in effect when RESET INTEGRAL is selected, should be that which yields the highest on-scale current reading.

- 3. [RESET INTEGRAL]: This key causes the final current integral, in coulombs, to be displayed. At the same time the coulometer is reset to zero and the initial scaling factor, as set by the selected Current Range, is established. **Note:** The reset state is maintained for three seconds or until the [RESET INTEGRAL] key is released, whichever is longer. At that point charge accumulation begins again.
- 4. [SET I OFFSET]: This key allows one to specify a current that is internally subtracted from the cell current. The offset is specified in units of current, and the offset range is ±2 times the full-scale range in effect at the time I OFFSET is set. Note that the offset function only affects the level at the OUTPUT connector (identically the Y Axis Recorder Output). It is functional for all three output modes (LINEAR, LOG, and COULOMBS). The displayed cell current is not affected, unless Override 10 (see Section 5.2.4) is placed in the YES state.

#### 5.3. Rear Panel

In addition to the ac power/fuse assembly (Chapter 2), there are numerous connectors and two switch assemblies located on the rear panel (Fig. 8). A brief description of each follows. Pinouts for the multi-pin connectors are provided in Appendix A.

**Warning!** For operator and equipment safety, the power should be off when connecting or disconnecting cables.

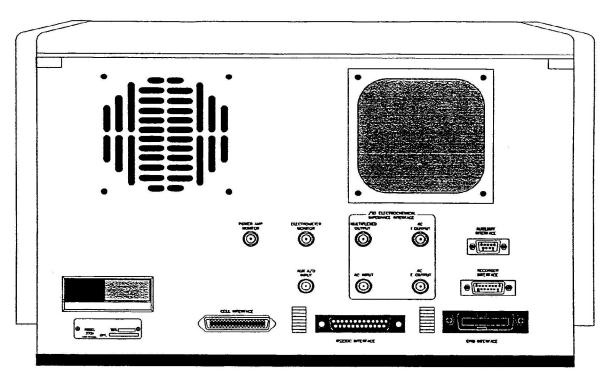


Fig. 8. Model 273A Rear Panel.

#### 5.3.1. Cell Interface

The cell cable mates with this 36-pin connector. The connections include shields and grounds, the electrode connections, and power for the Differential Electrometer Assembly. The hard-wired cable from the Differential Electrometer Assembly mates with this connector.

Warning! To prevent dangerous, possibly lethal electric shock, always have the front-panel cell enable switch in the off position when handling the clip leads at the end of the electrometer assembly. Voltages as high as ±100 V at currents as high as 1 A could be present.

#### 5.3.2. Recorder Interface

This 15-pin D-type connector is provided for interfacing between the Model 273A and a Princeton Applied Research RE0150 or RE0151 recorder. Connections include a Y-axis drive (same as the front-panel OUTPUT signal), an X-axis drive (same as the front-panel E MONITOR signal), and a contact closure for the Pen Up/Down function.

Two different recorder cables are available. C174 connects directly from the RECORDER INTERFACE connector to the interface connector on either the RE0150 or RE0151 recorder. With this cable installed, the OUTPUT signal drives the Y-axis, the E MONITOR signal drives the X-axis, and the pen closure requirements are provided for.

For operation with other recorders, or with an RE0150 or RE0151 recorder in applications where the normal X- and Y-axis drive signals are to be reversed (corrosion), cable C178 is recommended. One end of this cable has a connector that mates with the Model 273A's RECORDER INTERFACE connector. At the other end are two pairs of banana plugs and two leads terminated in spade lugs that provide the pen-drop contact closure. One pair of banana plugs provides the OUTPUT signal, the other the E MONITOR signal. Either can be connected to either the X-axis or Y-axis inputs of the recorder.

One of the two terminals of each banana plug pair has a small tab and is labeled "ground." This "ground" terminal connects to the recorder terminal (X or Y as appropriate) marked "-" (Houston) or LO (Hewlett Packard). The recorder terminal labeled GROUND on Houston recorders is not used in operation with the Model 273A.

A relay contact closure inside the Model 273A is brought out to the pen drop leads. These leads float with respect to ground. Many recorders have a pen-drop circuit configured such that the pen drops when the closure occurs.

#### 5.3.3. Auxiliary Interface

Various miscellaneous functions are provided by this connector, including.

- 1. Ground.
- 2. +5 V (20 mA maximum; sufficient for pullups).
- 3. Signals for interfacing to the Model 303A SMDE (PURGE, STIR, and DISLODGE).
- 4. Various inputs and outputs controlled by commands applied via the GPIB or RS-232C ports.

See Appendix A for more detailed information.

#### 5.3.4. Power Amp Monitor

The output of a 10:1 attenuator connected to the Counter Electrode potential is provided at this connector. The output impedance is 1  $k\Omega$ .

#### **5.3.5.** Aux A/D Input

This high-impedance (>10 $^{9}$   $\Omega$ ) input provides an external input to the A/D Converter. This input allows users to track any experimental variable that has an analog voltage output in the range of  $\pm$  10 V. For example, with the appropriate ancillary instrumentation (not available from Princeton Applied Research), a user might track temperature, pH, optical absorbance, or mechanical displacement. One specific use for this input is in making galvanic corrosion measurements with the Model 332 Corrosion Measurement System. Note that there is no way of reading the applied level from the front panel. A reading can be initiated only via commands applied to the rear-panel RS232C or GPIB ports.

#### 5.3.6. Electrometer Monitor

The output of the differential electrometer is available at this connector. This output is NOT corrected for  $R_{\it U}$  compensation (unlike the front-panel E MONITOR Output). The output impedance is 1 k $\Omega$ . Note that this potential is inverted in sign with respect to the applied cell potential.

## 5.3.7. AC I Output

This connector, used only in units equipped with the 273A/92 Electrochemical Impedance Interface option, outputs the ac current signal from the test cell. The signal is controlled by commands from an external computer via the GPIB or RS-232C port (refer to the *Model 273A Remote Programming Command Handbook*).

#### 5.3.8. AC E Output

This connector, used only in units equipped with the 273A/92 Electrochemical Impedance Interface option, outputs the ac voltage signal from the test cell. The signal is controlled by commands from an external computer via the GPIB or RS-232C port (refer to the *Model 273A Remote-Programming Command Handbook*).

## 5.3.9. Multiplexed Output

This connector is used only in units equipped with the 273A/92 Electrochemical Impedance Interface option. Either a current or voltage signal can be output from this connector as determined by commands from an external computer via the GPIB or RS-232C port (refer to the *Model 273A Remote-Programming Command Handbook*). Multiplexing is accomplished by alternating the operand of the MIE command between "1" and "2" on successive iterations.

Note: This output is intended for use with Princeton Applied Research electrochemical impedance systems.

#### 5.3.10. AC Input

This connector, used only in units equipped with the 273A/92 Electrochemical Impedance Interface option, accepts a sine wave input from an external oscillator, such as that supplied by a Princeton Applied Research lock-in amplifier. The nominal frequency range is  $50 \,\mu\text{Hz}$  to  $100 \,\text{kHz}$ , although this is dependent on cell impedance. Input voltage must be no greater than 5 volts rms. This signal can be used to modulate the signal sent by the Model 273A to the cell, and can be attenuated as desired. It is controlled by commands from an external computer via the GPIB or RS-232C port (refer to the *Model 273A Remote-Programming Command Handbook*).

# 5.3.11. 273A/92 Electrochemical Impedance Interface Option

The 273A/92 Electrochemical Impedance Interface option (previously called the AC Interface option) is intended primarily for use with Princeton Applied Research electrochemical impedance systems, but can also be used for ac voltammetry applications. This option is designed to allow superposition of an externally generated ac excitation signal on the dc signal generated by the Model 273A.

The externally generated ac signal is input at the AC INPUT connector and is combined with the signal generated by the Model 273A before it is applied to the cell. The /92 option board has three output connectors (AC I OUTPUT, AC E OUTPUT, and MULTIPLEXED OUTPUT. Software commands control both the mode of output (I, E, or multiplexed) as well as the type of signal applied (full ac + dc signal or ac signal only). The *Model 273A Remote-Programming Command Handbook* describes the computer commands used with the Model 273A/92 option.

#### 5.3.12. RS-232C Interface

This connector and the associated switch assembly are used to establish serial RS-232C communications between the Model 273A and a host computer. Instructions for remote operation are given in the separately bound *Model 273A Remote-Programming Command Handbook*. The adjacent switch assembly sets the user-controlled RS-232C parameters. See Appendices A and D of the *Command Handbook* for additional information.

#### 5.3.13. IEEE-488/GPIB Interface

This connector and the associated switch assembly are used to establish communications between the Model 273A and a host computer via the GPIB port (IEEE-488). Instructions for remote operation are given in the separately bound *Model 273A Remote-Programming Command Handbook*. The adjacent switch is used to set the GPIB address and TEST ECHO status, and allows for selection of the terminator. See Appendices B and C of the *Model 273A Command Handbook* for detailed information.

## 5.4. Differential Electrometer

The electrometer (Fig. 9) acts as the interface between the Model 273A and the reference electrode. A cable runs from the Electrometer Housing to the CELL INTERFACE connector at the back of the Model 273A.

The various leads, switches, and jacks on the front of the housing are briefly discussed in the following paragraphs.

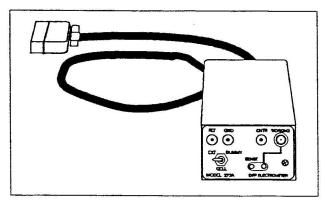


Fig. 9. Electrometer.

- 1. REFERENCE ELECTRODE JACK: The reference electrode plugs directly into this white jack. Several suitable electrodes are available from Princeton Applied Research.
- 2. CELL SWITCH: This switch has two positions, DUMMY and EXT. Normal operation occurs in the EXT position. In the DUMMY position, a 100 k $\Omega$ , 1% resistor inside the Electrometer Housing is substituted for the cell. The feature is useful for determining that the Model 273A is functioning correctly.

Note that this is a locking-type toggle switch. To change the setting, it is necessary to grasp the "bat" and pull it outwards a millimeter or two to release the catch.

Warning!

This switch provides no protection against the possibility of dangerous potentials being present at the cell cable leads. These leads can be handled safely only when the front-panel cell enable switch is in the off position.

3. SENSE and WORKING ELECTRODE JACKS: These two jacks are used together to give the operator control of the sense function. The upper "gray" jack is internally connected directly to the reference input of the Electrometer Amplifier. The lower "green" jack is connected internally to the adjacent Working Electrode lead, which ends in a clip having a green insulating boot.

In operation, the working electrode of the cell must be returned to the reference input of the Electrometer Amplifier. The user can do this in either of two ways. If error due to IR drop in the working electrode lead is not a consideration, simply connect the jumper (shorting plug) from the Sense jack (gray) to the Working Electrode jack (green) immediately below it.

When working at high currents, it may well be important to eliminate IR drop error resulting from the resistance of the working electrode lead and alligator clip. To do so, connect a lead from the Sense jack (gray) directly to the Working Electrode at the cell. Be sure to clip to the electrode and not to the clip at the end of the working electrode lead. Attaching the sense lead to the clip at the end of the working electrode lead will still leave the IR drop in the clip itself as a source of error. Sensing at the cell also prevents errors due to inductance in the working electrode lead. This inductance can cause significant phase shifts.

Sensing at the electrometer is convenient and yields good results in many applications. Sensing at the cell gives faster speed and freedom from errors due to IR drop in the working electrode lead. As a general rule, sensing at the cell is recommended in applications where the current will exceed 100 mA or where the equivalent cell impedance is less than 5  $\Omega$ .

What this comes down to is that the user must make a decision in each application where the electrometer is being used. If the decision is to ignore IR drops in the working electrode lead (certainly reasonable at low current levels), simply connect the shorting plug between the two jacks. If the decision is to eliminate this source of error, simply bring a lead from the Sense jack directly to the working electrode. One connection or the other should be made; never both.

In galvanostatic operation, one could conceivably choose not to monitor the potential with a reference electrode and so not use the electrometer. Where this is the case, no harm would come by operating with the Sense jack open. However, it is just as easy, and preferred, to install the jumper. If the potential is monitored, the same remote (cell) versus local (electrometer) sense considerations apply. The measured potential is affected by the iR drop.

4. ELECTRODE LEADS: There are three leads, each terminated in a clip. The leads can be readily identified by the color of the clip's insulating boot. Green identifies the Working Electrode and red the Counter Electrode.

Warning! Potentials as high as ±100 V could be present at the counter electrode lead's clip. For safety, always have the front-panel cell enable switch in the off position when handling the electrometer leads.

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.

Black identifies the ground lead. It can be used to ground a piece of peripheral apparatus, such as an electrostatic shield. Usually, this lead isn't used. TAKE CARE THAT THE ELECTROMETER LEAD CLIPS NOT SHORT TOGETHER. Because the ground lead is often unused, it tends to be overlooked, making the possibility of an accidental short involving this lead more of a problem than for the other two.

## 5.5. Model 273A Standard Environmental Conditions

## 5.5.1. Operating Conditions

This equipment shall meet all specified performance criteria when subjected to any natural combination of stress.

- A. Input Voltages
  - 1. 100/120 volts AC 50 to 60 Hz.
  - 2. 200/240 volts AC at 50 to 60 Hz.
- B. Ambient Temperature: From 10°C to 40°C
  - 1. The instrument shall operate from 10°C to 40°C but may not meet some temperature related specifications.
  - 2. The instrument shall operate from 20°C to 30°C and meet all its specifications over this range.

- C. Relative Humidity from 5% to 85% non-condensing.
- D. Altitude from -500 to 9000 feet relative to sea level

## 5.5.2. Non-Operating Conditions

This equipment shall exhibit no significant deterioration of performance after exposure to any natural combination of stress in 2.1 through 2.3 in a non-operating condition.

- A. Ambient Temperature range from 6°C to 70°C.
- B. 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.
- C. Altitudes to 50,000 feet.

#### 5.5.3. International Standards

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

- A. BS EN55022 (1987), Class B
- B. BS EN50082 (1992):
  - 1. IEC 801-2:1991
  - 2. IEC 801-3:1994
  - 3. IEC 801-4:1988
- C. BS EN61010-1 (1995), Installation Category II, Pollution Degree 2.

# **APPENDIX A. PINOUTS**

# A.1. Cell Interface (36 pins)

PIN	FUNCTION
1, 19	Counter Electrode
2,20	Counter Electrode Shield
4	Electrometer Output*
6, 8, 24, & 26	I / E Guard (Driven)
7, 25	I / E Input
10	+24 V (100 mA maximum)
12	-24 V (100 mA maximum)
14	Dummy Cell Sense
22	Electrometer Shield (gnd)
28, 29	Electrometer Ground
30	External Clip Ground
32	Digital Ground

<sup>\*</sup>Electrometer Output Potential = 15 x ( $E_{ref}$  -  $E_{working}$ )

# A.2. Recorder Interface (mating connector DP15P)

PIN	FUNCTION
1	ground
2	E MONITOR
3	ground
4	OUTPUT
5	ground
6	ground
7	ground
8	pen drop relay contact
9	pen drop relay contact
10	ground
11	ground
12	ground
13	ground
14	ground
15	ground

**Notes:** The E MONITOR and OUTPUT lines are driven by the same signals as are present at the front-panel E MONITOR and OUTPUT connectors.

When operating under front-panel control, the Pen-Drop relay contacts close when [START] or [CONTINUE] is pressed. Pressing [STOP] or [HOLD] causes the contacts to open. Under computer control (RS-232C or GPIB port), the pen contact closure is controlled by the PEN command.

# A.3. Auxiliary Interface (mating connector DB9S)

This connector provides several functions only available when the Model 273A is being controlled from an external computer via the RS-232C 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 307 Interface.

This accessory allows the Model 273A to be used in conjunction with the Model 303A Static Mercury Drop Electrode by providing three cables (all hard-wired to the Model 307) to make the necessary connections. One cable connects to the Model 273A AUXILIARY INTERFACE connector, the second connects to the Model 273A CELL INTERFACE connector, and the third connects to the Model 303A INPUT connector. A 0.1 A fast-blow fuse is provided in the Counter Electrode line. The Model 307 requires externally supplied +24 V at 1 A.

PI N	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 logic 0 is applied to the EXT. TRIG line. If the command WFT 1 is applied, operation will resume when a logic 1 is applied to the EXT. TRIG line.
3	DISPENSE	In applications where the Model 273A is being used with a Model 303A via a Model 307 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 <i>Model 273A Remote-Programming Command Handbook</i> ).
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 Interrupt 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 Comp. is active, this line will go high about 3 $\mu$ s before the start of the current interruption and will go low about 7 $\mu$ s after the start of the current interruption. This pulse can be used to trigger an osciloscope so that the IR Current Interrupt waveform can be easily monitored at the rear-panel ELECTROMETER MONITOR connector.
8	PURGE	When operating a Model 273A with a Model 303A SMDE via a Model 307 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 <i>Model 273A Remote Programming Command Handbook</i> ).

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

# A.4. IEEE-488 GPIB Interface

PIN#	FUNCTION
1	DI01
2	DI02
3	DI03
4	DI04
5	EOI
6	DAV
7	NRFD
8	NDAC
9	IFC
10	SRQ
11	ATN
12	SHIELD
13	DI05
14	DI06
15	DI07
16	DI08
17	REN
18	GND 6
19	GND 7
20	GND 8
21	GND 9
22	GND 10
23	GND 11
24	LOGIC GROUND

# A.5. RS 232C Interface

PIN	FUNCTION	COMMENT
1	Earth Ground	Ties the chassis of the 273A to that of the computer.
2	Transmit Data	The 273A transmits on this line. It must connect to the computer connector pin that receives serial data.
3	Receive Data	The 273A 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 273A, that is, it is always at a positive logic level (+12 V). As a result, the computer continuously receives the message that the Model 273A is ready to receive a character.
5	Clear to Send	The computer controls the 273A via this line. To enable the 273A to transmit, the line is placed at the positive logic level (+3 V to +12 V). To hold off transmission by the 273A, the line must be at the negative logic level (-3 V to -12 V). Once this line goes positive, data transfer, if initiated by an appropriate command, will proceed rapidly. This line is pulled up to + 12 volts so that, if it is left unconnected, the 273A 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 273A should interconnect with the logic ground line of the computer.
8 -10	Unused	
11	+5 V	Unless +5 V (100 mA max) is needed by the connected apparatus, don't connect to this pin. In no case should this +5 V level be tied to the +5 V level in the computer. +5 V can be used to continuously assert the Clear-to-Send Line (pin 5), allowing the 273A to transmit at any time.
18	+12 V	
20	+12 V	+12 V (300 Ω in series)
25	-12 V	

Note that the 273A RS-232C 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-232C cable, also known as a null modem cable.

# APPENDIX B. RACK MOUNTING

The Model 273A can be mounted in a standard 19 inch relay rack by means of the Princeton Applied Research 1715-0206 Rack Mounting Kit. This kit consists of two special side panels with integral mounting flanges and four screws. To install the kit, proceed as follows.

- 1. Remove the side panels from the instrument. Each side panel is secured by four screws. *DO NOT DISCARD THESE SCREWS*.
- 2. Mount the side panels supplied with the kit. The flanges should go toward the front and turn outwards. Secure each panel with *SIX* screws. You will need the four screws supplied with the kit as well as the eight screws that became available when the original side panels were removed.

This completes the procedure. The 273A can then be mounted in any standard rack and secured with standard rack-mounting screws (not supplied). Because of the Model 273A's considerable weight, we strongly recommend that it be supported to prevent excessive stressing of the front panel. One way of providing the necessary support is to set the Model 273A on a rack-mounted shelf or drawer.

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