SECTION III
OPERATING INSTRUCTIONS

3.1 INTRODUCTION

Operation of the Model 362 Scanning Potentiostat is straightforward. The connections to the cell are made with the CELL pushbutton OFF. Then the controls are set as required to establish the desired operating mode and parameters. When satisfied that the system is ready, initiate the experiment by switching the CELL pushbutton ON. This section of the manual discusses the individual controls in greater detail. Static CONTROL E and CONTROL I modes are disscussed first, followed by some general discussions of operating considerations. Subsections 3.8 and 3.9 focus on the linear scanning modes of CONTROL E and CONTROL I operation.

3.2 FRONT-PANEL CONTROLS AND CONNECTORS

3.2A INITIAL POTENTIAL

This set of five digital thumb switches sets the controlled potential, current level, or ramp limits. NOTE: When the position of one of these switches is changed, there is a momentary deferral to the setting of the less significant switches before the new level is established. For example, if the switches were set to 2.000, and the setting were changed to 1.000, there would be an instant in the transition when the switches would have an output equivalent to a setting of 0.000. This might prove a problem in certain applications.

(1) CONTROL E: In this mode the INITIAL POTENTIAL value extends from −9.999 V to +9.999 V. Note that the limit of ±9.999 rests on the assumption that no signal is applied to the EXT IN input. If a signal is present, a more general limit applies, namely, that the algebraic sum of the INITIAL POTENTIAL setting plus the external input voltage cannot exceed ±9.999 V. In actual practice, the system may control slightly beyond the specified limits. At the point where the loop loses control, the CONTOL AMPLIFIER OVERLOAD lamp lights.

(2) CONTROL I: In this mode the INITIAL POTENTIAL control works in conjunction with the CURRENT Range pushbuttons to set the current level. The pushbuttons define the "full scale" of the current scanning limit. The actual controlled current level will be the product of the selected current range times the INITIAL POTENTIAL setting. for example, if the 1 mA CURRENT Range pushbutton were depressed with the INITIAL POTENTIAL control set to +1.500, the current would be maintained at a constant 1.500 mA. Note that it is perfectly possible to operate with a constant current greater than full scale, the controlled current in the example being 1.5X full scale. Full scale simply defines the current that gives 1 V out at the CURRENT MONITOR output. The limit is two full scales (exception: The 1 A range, where the limit is 1 A). Higher currents will trigger an overload indication. Thus, in CONTROL I operation, the usable range of the INITIAL POTENTIAL control is limited to ±2.000, except on the 1 A range where the limit is ±1.000.

Note that the limit of ±2.000 or ±1.000, whichever applies, rests on the assumption that no signal is applied to the EXT IN input. If such an input is present, a more general limit applies, namely, that the algebraic sum of the INITIAL POTENTIAL setting plus the external input voltage cannot exceed ±2.000 or ±1.000, whichever applies. The EXT IN input signal should always be a potential, even though current is the controlled parameter.

3.2B AC

This is the AC power switch. The switch is self-illuminated, indicating immediately whether the instrument is powered.

3.2C MODE

This pushbutton switch allows the operator to select either CONTROL E (*pushbutton released*) or CONTROL I (*pushbutton in*) operation.

3.2D EXT IN

The EXT IN input connector (*EXTERNAL INPUT*) allows the user to apply external control signals that, together with the INITIAL POTENTIAL control, set the potential or current level. The applied signal is summed algebraically with the setting of the INITIAL POTENTIAL control. For example, during operating in the CONTROL E mode with an INITIAL POTENTIAL setting of +1.000, +1 V applied to the EXT IN connector establishes the working electrode potential at +2 V with respect to the reference electrode. The situation is similar in CONTROL I operation. An INITIAL POTENTIAL of +0.500 combined with +1 V applied to the EXT IN connector yields a current of 1.5X the selected current range.

3.2E CELL SWITCH, CONNECTOR & CABLE

The Cell Cable (6020-0168) connects to the front-panel CELL connector. At the other end of the cable are four leads, three of which terminate in color-coded alligator clips, and one of which terminates in a pin jack. In both CONTROL E and CONTROL I operation, the red clip connects to the counter electrode and the green clip connects to the working electrode. The reference electrode output plugs into the white pin jack. The black clip is a ground. Although not *required* for any of the Model 362's operating modes, it is available if needed for some special purpose, such as to supply ground to a shielding screen surrounding the cell. If the ground lead isn't used, take care not to let it short against any of the other clips. The user may find it useful to read EG&G PARC Application Note TN117, GROUNDING AND SHIELDING IN ELECTROCHEMICAL INSTRUMENTATION - SOME BASIC CONSIDERATIONS.

The CELL pushbutton, located directly above the connector, determines whether the CONTROL AMPLIFIER is connected to the cell. In the OFF position (*released*), the CONTROL AMPLIFIER is disconnected from the cell and there is zero cell current, allowing the cell connections to be made safely. In the ON position (*in*), the CONTROL AMPLIFIER is connected to the cell, making contact with the cell cable clips potentially hazardous. For this reason, the cell cable clips should only be handled when the CELL pushbutton is in the OFF position.

3.2F METER SWITCH AND METER

The panel meter indicates either current or voltage, as selected by the METER pushbutton. When the METER pushbutton is released, the meter indicates the working electrode current, which is read from the upper meter scale. The polarity of the current indication is the same as that at the CURRENT MONITOR output. Full-scale meter deflection corresponds to a current equal to the selected current range.

When the METER pushbutton is depressed, the meter indicates the potential of the working electrode with respect to the reference electrode. Potential readings are taken from the lower meter scale, which has a range of ±5 V. The polarity of the meter indication will be opposite that at the POTENTIAL MONITOR output. The polarity conventions for the Model 362 are indicated in the specifications and in Figures I-3 and I-4.

3.2G CURRENT RANGE PUSHBUTTONS

This set of seven pushbuttons sets the current range in both modes. However, the significance of the current range differs from one mode to another, as follows.

(1) CONTROL E: The potential is controlled in this mode. The current assumes whatever value is appropriate for the various system parameters. The CURRENT RANGE pushbuttons give the operator the capability of making accurate current measurements over a range extending from a full scale of 1 µA to a full scale of 1 A. For best accuracy, one should choose that current range which provides the highest on-scale meter indication. If the current range selected is too big, for example, 1 A with a 1 mA current, the resultant CURRENT MONITOR output and meter indication (meter in I mode) will be small, and the error, as a fraction of the signal level, will be large. If the current range is too small, for example, 1 mA with a 10 mA current, the current measuring circuits will overload (the CURRENT OVERLOAD indicator lamp lights at two times full scale). The potential control is maintained despite the overload in the current-monitoring circuits.

(2) CONTROL I: The CURRENT RANGE pushbuttons have a two-fold function in this mode. First, together with the INITIAL POTENTIAL control (and any EXT IN signal) they determine the magnitude of the controlled current. (In the linear scaning mode the setting of the SCAN RATE switch is also a factor in determining the controlled current.) The INITIAL POTENTIAL setting is multiplied by the current range to determine the current level. For example, with an input of 1.000 V, and a CURRENT RANGE setting of 1 mA, the current will be maintained at 1 mA. Second, the pushbuttons set the meter sensitivity (meter in I mode), the same as in Control E operation.

Again, for highest accuracy, it is desirable that the current range provide the highest on-scale meter indication. For example, suppose one wanted to control the current at 1 mA. One way would be to set the INITIAL POTENTIAL to 1.000 and select the 1 mA current range. The product would be 1 mA, the desired current level, and the meter sensitivity would be full-scale deflection with 1 mA of cell current. Alternatively, the same current level could be established by setting the INITIAL POTENTIAL to 0.100 and selecting the 10 mA current range. As before, the product would be 1 mA and the current would be controlled at that level. However, with a current range of 10 mA and a 10% of full scale setting, the inherent noise and drift of the instrument will be larger than with a 1 mA range and a 100% of full-scale setting. The reserve range at 10% of full scale, however, would extend to twenty times the controlled current (ratio of 20 mA to 1 mA), which could be a factor to consider in certain situations.

3.2H POTENTIAL MONITOR OUTPUT

In both modes, the signal provided at this output is a voltage equal to the reference electrode with respect to the working electrode. The meter, on the other hand, indicates the potential of the working electrode with respect to the reference electrode. The two potentials are equal in magnitude but of opposite polarity. The output range is ±9.999 V and the output resistance is a few ohms (the current capability, however, is limited; the load resistance should not be less than 2 kΩ at full output).

3.2I CURRENT MONITOR OUTPUT

In both modes, the signal provided at this output is a voltage that varies directly with the cell current. The polarity is the same as the meter indication (I mode) and a full-scale meter indication corresponds to 1 V out. (The polarity convention is stated in the specifications). Linear operation is assured up to 2 V out (two times full scale), except on the 1 A range, where the limit is 1 V out. Although the output resistance is only a few ohms, the load resistance should not be less than 2 kΩ at full output.

3.2J RAMP OUT

The RAMP OUT output signal is a voltage equal in magnitude (but opposite in polarity) to any deviation between the INITIAL POTENTIAL and the voltage applied to the cell as a result of the scanning modes. (In the static modes there is no deviation and the RAMP OUT level is 0 V.) In constrast, the POTENTIAL MONITOR output signal is a voltage equal in magnitude (but opposite in polarity) to the sum of the INITIAL POTENTIAL and any voltage added to or subtracted from it as a result of the scanning modes. (In the static modes, no ramping voltage is applied to the cell, so the POTENTIAL MONITOR signal is simply equal in magnitude and opposite in polarity to the INITIAL POTENTIAL setting.) The load resistance attached to the RAMP OUT output should be greater than 2 kΩ.

3.2K OVERLOAD INDICATIONS

There are two OVERLOAD lights, one labeled CURRENT and the other CONTROL AMPLIFIER. The significance of an overload indication depends on the operating mode, as follows.

(1) CONTROL E A CURRENT OVERLOAD indication means that the working electrode current is greater than two times the selected CURRENT Range. It does not mean that the loop is out of control. For example, suppose the controls were set to establish a potential of +0.752 V, and that, under the established condition, a cell current of 0.5 mA occurred. If the selected CURRENT Range were 1 µA, 10 µA, or 100 µA, the current monitoring circuits would be driven beyond 2X full scale, causing a CURRENT OVERLOAD indication. Although the CURRENT MONITOR Output and meter indication (I display mode) would be invalid, the cell would still be controlled at +0.752 V, as programmed.

On the other hand, a CONTROL AMPLIFIER Overload indication means unequivocally that the loop is out of control, that is, the potential of the working electrode with respect to the reference electrode is NOT that programmed. This could result from a connection error (see Subsection 3.4), an electrode problem, or an unacceptably high solution resistance. It will also occur if the cell current exceeds 1 A. (Currents higher than 1 A cause the CONTROL AMPLIFIER Overload indicator to light, a sign that control has been lost.)

(2) CONTROL I: A CURRENT OVERLOAD indication means that the working electrode current is greater than two times the selected Current Range. Since it is the current that is the controlled parameter in this mode, if the cell connections have been made properly, about the only way this can happen is if the setting of the INITIAL POTENTIAL, the ramp voltage, or the potential applied to the EXT IN input are such as to program a current higher than two full scales. A programmed level of 2.000 would result in the current being controlled at twice the selected current range. Higher settings would result in a CURRENT OVERLOAD indication.

A CONTROL AMPLIFIER OVERLOAD indication means that the compliance of the amplifier has been exceeded (or that the current exceeds 1 A). In CONTROL I operation, this would normally only occur with a high resistance solution or an open cell connection.

3.3 LINE CORD AND FUSE ASSEMBLY (REAR PANEL)

This assembly allows the operator to readily change the line fuse. it also has provision for optimizing the Model 362 power circuits to the prevailing line voltage over a wide range. These operations are discussed in detail in Section I.

3.4 INSTALLATION

Generally speaking, the Model 362 can be operated on a typical laboratory bench in a typical laboratory environment without difficulty. Usually the experiment to be controlled would be located to one side or the other on the same bench. Be sure to allow at least six inches (15 cm) between the rear-panel heat sink and the back of the bench. This much space is required to assure reliable operation in a high-current application. If the Model 362 is operated at a high current in a closed area, such as rack cabinet, it is advisable that forced air cooling of the rear-panel hat sink be used to assure reliable operation. Such cooling can be implemented by means of an external fan directed at the rear panel. Some equipment cabinets have built-in forced-air cooling adequate for this purpose.

Other than to provide the instrument with AC power as described in Section I, no special hookups are required. If the experiment is one that requires deoxygenation of the electrolyte, the necessary additional equipment and oxygen-free gas will have to be furnished (see EG&G PARC Electrochemical Accessories catalog).

Connections to the cell are made using the cable furnished with the instrument. One end of the cable terminates in a connector that mates with the front-panel CELL connector. At the other end are four leads. Three are color-coded leads, each terminated in a spring-loaded clip. The fourth one is terminated in a pin jack. The cell connections are made as follows.

WARNING!

FOR OPERATOR SAFETY, DO NOT INSTALL THE CABLE, CONTACT THE CABLE LEADS, OR MAKE THE CELL CONNECTIONS UNLESS THE MODEL 362 CELL PUSHBUTTON IS IN THE OFF (*released*) POSITION.

(1) RED LEAD: Connects to the counter (auxiliary) electrode in both modes.

(2) GREEN LEAD: Connects to working electrode in both modes.

(3) BLACK LEAD: Available as a source of ground potential. This is not a *required* connection for any operating mode.

(4) PIN JACK: Connects to the reference electrode. reference electrodes manufactured by EG&G PARC are furnished with a connecting lead that directly plugs into this pin jack. Note that, whereas a reference electrode is *essential* in CONTROL E operation, in CONTROL I operation a reference electrode is not necessary for control, but only to provide a valid *E* meter indication and POTENTIAL MONITOR output.

3.5 CONNECTION ERROR DETECTION

Although connection errors are not likely, they could occur. Most commonly the problem is that for one reason or another one of the clips has been removed from an electrode, and then forgotten. Subsequent attempts to establish operation will then prove unsuccessful. An electrode can be "open" for other reasons as well. Failure to fill the cell or the reference electrode with the appropriate solutions will give open-electrode indications. Bubbles on an electrode can also impair its performance. Thus, any time an experiment doesn't seem to be behaving properly, the first step should be to simply look at the cell and verify that the connections have been properly made and that the cell has been properly assembled and filled. If everything looks all right, try running the experiment and check the indications obtained against the following table:

MODE	OPEN ELECTRODE	I METER	E METER	POTENTIOSTAT OVERLOAD	CURRENT OVERLOAD
Cont. E	Counter	"0"	O.K.	ON	OFF
Cont. E	Reference	Off Scale	Off Scale	ON	ON
Cont. E	Working	"0"	O.K.	OFF	OFF
Cont. I	Counter	"0"	O.K.	ON	OFF
Cont. I	Reference	O.K.	Off Scale	OFF	OFF
Cont. I	Working	"0"	O.K	OFF	OFF

Table III-1. OPEN ELECTRODE INDICATIONS AS A FUNCTION OF OPERATING MODE

3.6 CONTROL E OPERATION

In Control E operation, the potential of the working electrode with respect to the reference electrode is controlled at the potential set with the INITIAL POTENTIAL control, together with any voltage added or subtracted by the scanning modes or by the EXT INT input. For example, to control the working electrode at +0.755 V, the user would simply set the controls to +0.755. The voltage range over which control can be maintained is ±9.999 V.

It is important to understand that the potential established is with respect to the reference electrode and not with respect to ground. Both the reference and working electrodes are "off ground" when driven by a Model 362. The potential of the working electrode with respect to the reference electrode can be read from the panel meter if the meter £ mode is selected.

Alternatively, a more accurate measurement can be made with an external voltmeter connected to the POTENTIAL MONITOR output. The potential provided at this output is that of the reference electrode with respect to the working electrode, that is, the inverse of the parameter of interest.

The following procedure should prove suitable for establishing operation in the CONTROL E mode.

(1) Prepare the Cell. Be sure the electrodes are immersed in the solution. Also, check that the reference electrode is properly filled.

(2) With the Model 362 AC power switch and CELL pushbutton both set to OFF, connect the cable. One end mates with the front-panel CELL connector. The other end connects to the electrodes at the cell. The red lead connects to the counter electrode and the green lead connects to the working electrode. The output lead of the reference electrode connects to the pin jack. The black (ground) lead is normally unused, although it could be used to establish a shield or other piece of peripheral apparatus at ground potential. If the black lead is unused, take care that it doesn't short to any of the other active circuit elements.

Two-electrode operation is also possible. In this configuration there is no reference electrode. The red lead and the white lead are shorted together and connected to the counter electrode. The green lead connects to the working electrode. As in three-electrode operation, the black lead is available as a convenient source of ground potential.

(3) Set the AC power switch to the ON position.

(4) Set the INITIAL POTENTIAL control to the intended control potential. The potential of the working electrode with respect to the reference electrode will be controlled at the set value. (This will not happen until the CELL switch is set to ON, a later step.) If a control sgnal is to be applied to the EXT IN connector, make the necessary connections at this time. Bear in mind that the working electrode will be controlled at the sum of the external control potential plus that set with the INITIAL POTENTIAL control, and that this sum cannot exceed ±9.999 V for control to be maintained.

(5) Be sure the MODE pushbutton is in the CONTROL E *(released)* position.

(6) Check the METER pushbutton. It should be set to *E (in)* That way, when the CELL pushbutton is switched ON (step 8), there will be immediate verification of system control because the working electrode potential with respect to the reference electrode will be indicated. In *this* step, since the CELL pushbutton is still set to OFF, there is no counter electrode connection to the cell and the control loop is open. The reference electrode connection, however, is intact, with the result that *the meter displays the open circuit potential of the cell* (open-circuit working electrode with respect to reference electrode). This reading may or may not be an important parameter in the intended experiment.

(7) Select the 1 A CURRENT RANGE. Once the measurement is underway, a current range appropriate to the current level present can be quickly established.

(8) At this point the system should be ready. All that remains is to press the CELL pushbutton. When this is done, the panel meter will immediately indicate the closed-loop potential of the working electrode with respect to the reference electrode. This potential should be te same as the sum of the potential set with the INITIAL POTENTIAL control plus any external input. If the EXT IN input is not used, the meter indication will equal the INITIAL POTENTIAL setting, telling the operator that the control loop is functioning properly. An equal potential of opposite polarity will be available at the POTENTIAL MONITOR output. If the potentiostat is not controlling, the CONTROL AMPLIFIER OVERLOAD indicator will light. (A cell current greater than 1 A will also cause this lamp to light.)

(9) Having verified potentiostatic control, establish the *I* meter mode (release the METER pushbutton). Then select successively lower current ranges until the maximum on-scale meter indication is obtained. Full-scale current equals the selected current range, allowing the cell current magnitude and polarity to be quickly and easily read. A proportional voltage of the same polarity is available a the CURRENT MONITOR output.

This procedure should prove adaptable to most real measurements. Potentiostatic control of the cell will be maintained until terminated by the operator (CELL pushbutton to OFF). Because of the switching transition characteristics of the INITIAL POTENTIAL control, users are advised to use caution in changing the setting of this control once CONTROL E operation is established. In most instances, when the setting of the most significant switch is changed, there is a momentary deferral to the less significant switches before the new level is established. For example, if the switches are set to 3.000 V, and the setting is changed to 2.000 V, there will be an instant in the transition when the switches will have an output corresponding to a setting of 0.000 V. This might prove a problem in certain applications.

3.6A IR COMPENSATION

The Model 362 includes a set of front-panel controls to compensate for the cell resistance between the reference electrode and the working electrode. These controls, labeled IR COMPENSATION, are used in the CONTROL E mode only to apply positive feedback to the control amplifier. The feedback signal is a voltage produced by a the current (I) in the resistance between the reference and working electrodes (R), hence the notation *IR compensation*. The instrument's control loop automatically compensates for cell resistance between the counter electrode and the reference electrode.

The front-panel controls consist of a latching ON-OFF toggle switch and an ten-turn potentiometer with a high-resolution dial. The toggle switch should be OFF whenever the instrument is not in the CONTROL E mode or whenever IR compensation is not desired. The amount of feedback varies with the potentiometer's dial setting. With the dial fully clockwise (indicating 10.00) the feedback is 100%. The feedback voltage is a portion of the CURRENT MONITOR signal.

3.6B SETTING THE IR COMPENSATION CONTROLS

The exact amount of positive feedback required in a given system is a function of many factors, including the system impedance, the uncompensated solution resistance and the nature of the signal being applied to the system. Because of the number of different factors involved, it is difficult to find a universally valid procedure for setting the IR compensation. Nevertheless, here's a general method that usually yields good results.

This method involves pulsing the working electrode in potential regions where no faradaic processes are occurring, while monitoring the current vs. time behavior of the system at the CURRENT MONITOR output. The ability of the working electrode to follow an applied square-wave signal is a function of the solution resistance, R, between the working and reference electrodes, and of the double-layer capacitance, C, of the working electrode. This RC combination slows the response of the working electrode to the applied square wave. Positive feedback IR compensation increases the potential so that the solution RC can be quickly charged. The procedure for setting the IR compensation follows:

(1) Prepare the cell in the usual manner. To prevent faradaic current, the potential applied with the INITIAL POTENTIAL control should be well below the half-wave potential of the reaction of interest.

(2) Set up an external square-wave generator to provide a small (10-50 mV pk-pk) square wave at 1 kHz or lower, and connect the square wave to the front-panel EXT IN connector. Note: many oscilloscopes are equipped with an internal calibrator that provides a square-wave signal at either 1 kHz or 60 Hz. Such a signal can be used to adjust the IR compensation.

(3) Connect an oscilloscope to the CURRENT MONITOR connector and adjust the oscilloscope to the current vs. time relationship of the electrode response.

(4) Turn the IR COMPENSATION dial fully clockwise. Turn the IR COMPENSATION switch ON. Then begin rotating the dial clockwise while monitoring the CURRENT MONITOR signal with the oscilloscope.

(5) Adjust the dial for the smoothest fast-charging of the double-layer capacitance, as shown in Figure III-1. With too little compensation, the response will be slow and of low amplitude. As the compensation is increased, the amplitude of the observed waveform will increase and the charging time will become faster. Too much compensation results in a very high amplitude waveform with fast response and ringing that indicates the system is not well controlled. In many cases, it will even be possible to make the entire system oscillate completely out of control by setting the IR compensation too high. The correct setting is that which gives a response similar to that shown in the lowest curve of Figure III-1, that is, fast, but without ringing. If the IR compensation is too low with the dial fully clockwise, as may be the case in high-resistance solutions, operating with 100% positive feedback is the best you can do.

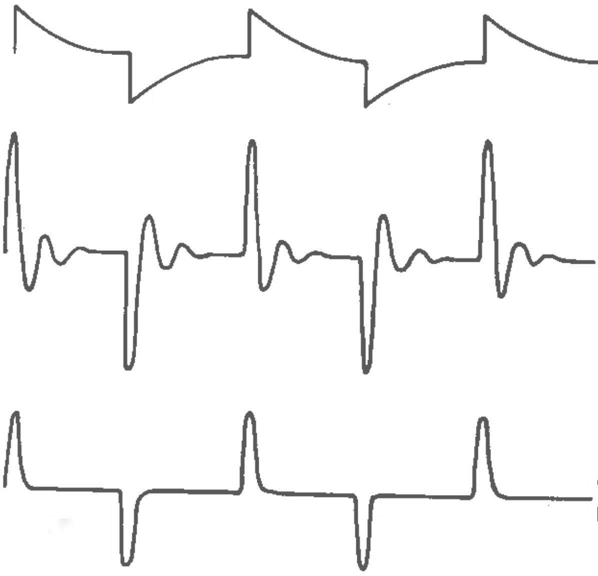

Figure III-1. CURRENT MONITOR WAVEFORMS DURING IR COMPENSATION PROCEDURE

IR compensation adjustment are critical in the sense that any change in the system (sensitivity, cell geometry, supporting electrolyte, etc.) will change the amount of compensation required. The IR compensation should always be adjusted with the CURRENT RANGE set to the intended operating range. If the CURRENT RANGE is changed in the course of the experiment, the IR compensation will have to be readjusted. Bear in mind that IR compensation has significance in CONTROL E operation only.

3.7 CONTROL I OPERATION

In Control I operation, the cell current is maintained at the level set by the INITIAL POTENTIAL control, acting in conjunction with the selected range, and, if applicable, the scanning function. The setting limits are ±2.000, that is, two times the full-scale CURRENT RANGE setting. As in Control E operation, the EXT IN function is active. If an external input is applied, it is summed with the INITIAL POTENTIAL setting to determine the control current level. The control range for the sum is the CURRENT RANGE setting, except on the 1 A Current Range where it is one full scale. An input of 1.000 V is required to establish a current equal to the selected range.

By furnishing the cell with a reference electrode connected in the same way as for Control E operation, a valid POTENTIAL MONITOR output will be available, and the potential of the working electrode with respect to the reference electrode can be read from the panel meter (*E* meter mode). This function is separate from the current control function, which does not require the presence of a reference electrode.

The following procedure should prove suitable for establishing Control I operation in many experiments.

(1) Prepare the cell. Be sure the electrodes are immersed in the solution. Also, check that the reference electrode, if one is to be used, is properly filled and free of bubbles.

(2) With the Model 362 AC power switch and CELL pushbutton both set to OFF, connect the cable. One end mates with the front-panel CELL connector. The other end connects to the electrodes at the cell. The red lead connects to the counter electrode and the green lead connects to the working electrode. If the cell includes a reference electrode, the output lead of the reference electrode should be connected to the pin jack. If the cell doesn't include a reference electrode, ground the pin jack (the black lead can serve as a convenient source of ground). With ground applied to the pin jack, the meter will indicate off scale in the *E* mode and the POTENTIAL MONITOR Output will go to full scale.)

It is also possible to connect the pin jack to the red lead, which in turn is connected to the counter electrode. This configuration allows two-electrode potential measurements (useful in battery and fuel-cell experiments).

(3) Set the AC power switch to the ON position.

(4) Select the desired CURRENT RANGE. Usually, the best range will be that which allows the highest setting of the INITIAL POTENTIAL control in establishing the current level. For example, in setting a current level of 1 mA, a current range of 1 mA (requiring an INITIAL POTENTIAL setting of 1.000) will usually be preferable to a current range of 10 mA (requiring an INITIAL POTENTIAL setting of 0.100).

(5) Set the INITIAL POTENTIAL control as required to establish the desired current. If a control signal is to be applied to the EXT IN connector, make the necessary connections at this time.

(6) Be sure the MODE pushbutton is set to CONTROL I (*in*).

(7) Be sure the METER pushbutton is set to display current (*released*).

(8) At this point the system should be ready. If the open-circuit cell potential is of interest, press in the METER pushbutton and read the potential from the panel meter. (Alternatively, record the potential at the POTENTIAL MONITOR Output.) When this is done, or, if the open-circuit cell potential is of no interest, press the CELL pushbutton to establish current control. The *I* Meter mode (METER pushbutton *released*) should be selected. The panel meter will indicate the actual current level, which should be that programmed. To monitor the working electrode potential (only possible if the system includes a properly connected reference electrode), simply select the *E* meter mode (METER pushbutton depressed) and read the potential from the panel meter. Alternatively, monitor the potential at the POTENTIAL MONITOR output with an external voltmeter; the potential at this output will be equal but opposite to that indicated by the panel meter. Again, the cell must be equipped with a reference electrode for the potential to be read at either the panel meter or the POTENTIAL MONITOR connector.

The preceding procedure should prove adaptable to most real measurements. Galvanostatic control of the cell will be maintained until terminated by the operator (CELL pushbutton OFF). Because of the switching transition characteristics of the INITIAL POTENTIAL control, users are advised to use caution in changing the setting of this control once CONTROL I operation is established. In most instances, when the setting of the most significant switch is changed, there is a momentary deferral to the setting of the less significant switch before the new level is established. For example, if the switches are set to 2.000, and the setting is changed to 1.000, there will be an instant in the transition when the switches will have an output corresponding to a setting of 0.000. This might prove a problem in certain applications.

3.8 LINEAR SWEEP SCANNING MODES

The instrument's linear sweep scanning modes produce a voltage (CONTROL E) or current (CONTROL I) output that varies with time, beginning at a level set through the front-panel INITIAL POTENTIAL selector and continuing to a level corresponding to the FINAL POTENTIAL SETTING. The SCAN RATE switch governs the rate of the scan. The procedure for selecting a scan rate depends upon whether the instrument is in the CONTROL E or CONTROL I mode. An internal SCAN RATE MULTIPLIER switch allows the operator to select scan rates at X10 the front-panel setting.

A bank of seven SCANNING CONTROL pushbutton switches controls the scanning options and parameters. These controls are located in a vertical row on the left-hand side of the front-panel (viewed from the front of the instrument). All but the REVERSE switch are latching, and during normal operation are either *IN* or *RELEASED*. Labels to the right of the switch bank indicate how each switch affects to the instrument's scanning modes.

As is the case with the non-scanning modes, the instrument's output is not applied to the CELL electrodes until the CELL switch is ON.

3.8A SCANNING CONTROL SWITCHES

$E_{INITIAL}$ (*released*): The $E_{INITIAL}$ position disables the scanning modes and returns the instrument's output to the CONTROL E or CONTROL I value selected through the INITIAL POTENTIAL setting. An exception to this is that in the CONTROL I CONSTANT I mode, releasing this button halts the current output.

RUN (*in*): This position initiates scanning toward the INITIAL POTENTIAL setting.

REVERSE (*in*): A momentary switch, REVERSE (*IN*) causes the output ramp to change direction. If the output were moving from the INITIAL to the FINAL POTENTIAL setting, for example, and REVERSE were pressed, the output would change direction and ramp back toward the INITIAL POTENTIAL setting before continuing.

HOLD (*in*): If the instrument is scanning and HOLD is pressed scanning halts, and the output changes to a DC level equal to the instantaneous magnitude of the output when button is pressed. When HOLD is *RELEASED*, scanning continues.

SINGLE (*in*): Pressing SINGLE selects SINGLE rather than MULTIPLE scanning. If STOP AT E_F is selected, the output ramps from the INITIAL POTENTIAL value to the FINAL POTENTIAL value and halts. If STOP AT E_I is selected, the output ramps first to the FINAL POTENTIAL, then ramps back to the INITIAL POTENTIAL before halting.

MULTIPLE *(released)*: Releasing this key instructs the instrument to ignore the STOP AT... selections and thus enables continuous scanning, which is halted only by operator intervention.

STOP AT E_F *(in)*: When *in*, this button selec s E_F, the FINAL POTENTIAL setting, as the endpoint for a single scan. This button has no effect when MULTIPLE scans are selected.

STOP AT E_I *(released)*: This button selects the endpoint of a single scan as E_I, the INTITIAL POTENTIAL setting. It is ignored if multiple scanning is selected.

LINEAR SCAN *(released)*: This button selects linear scanning (ramp function output) in either the CONTROL E or CONTROL I modes.

CONSTANT I *(in)*: Used while CONTROL I is enabled, This button instructs the instrument to apply a constant current to a cell or battery while cycling its potential between the INITIAL POTENTIAL and FINAL POTENTIAL settings (see Subsection 3.9). (Selecting CONSTANT I and CONTROL E simultaneously produces an output that is unpredictable and unrelated to other parameter and option settings.)

3.8B SCANNING MODE MONITOR OUTPUTS

While the instrument is in its scanning modes, it produces three output signals which can be connected to external monitoring apparatus, such as a recorder, or used as signal input to a complex control system. These signals are output on on three separate sets of binding posts. Each set includes a black binding post, nominally at chassis ground potential, which carries the signal's reference level.

The RAMP OUT signal is a voltage equal in magnitude and opposite in polarity to the voltage added to or subtracted from the INITIAL POTENTIAL as a result the scanning functions. The polarity of the RAMP OUT signal is opposite the polarity of the instrument's output (as shown on the panel meter) but is the same as the polarity of the voltage on the counter electrode. For example, if the INITIAL POTENTIAL is set for 0.000 V and the FINAL POTENTIAL is set for +5.000 V, the panel meter will display a voltage increasing from 0.0 to +5 V at a rate set by the SCAN RATE control. The corresponding voltage on the RAMP OUT binding posts will be 0.0 to -5 V (the green binding post with respect to the black).

The magnitude of the POTENTIAL MONITOR signal, on the blue binding post, is equal to the voltage of the working electrode with respect to the reference electrode. Thus, its limits are equal in magnitude and opposite in polarity to the INITIAL POTENTIAL and FINAL POTENTIAL settings. Like the RAMP OUT signal, its polarity is the same as that of the voltage on the counter electrode.

The CURRENT MONITOR signal, on the red binding post, is a voltage proportional to the cell current, where 1 V represents a full-scale CURRENT RANGE level. The polarity of this voltage matches that of the current as it is displayed on the front-panel meter. If the current exceeds approximately two times the full-scale current range value, the CURRENT OVERLOAD lamp will light. This is not of itself cause for alarm, as it simply means that the current is twice the full-scale meter value and that the voltage on the CURRENT MONITOR outputs may not be linear with respect to the cell current. Switching to a higher CURRENT RANGE should remedy the problem and bring the panel meter indication off full scale.

3.8C CONTROL E LINEAR SCANNING

The output of this mode is a voltage ramp applied to the working electrode with respect to the reference electrode. The ramp begins at a voltage level corresponding to the the INITIAL POTENTIAL setting and extends to the FINAL POTENTIAL setting. In the CONTROL E mode, the operation of the SCAN RATE switch is straightforward and allows selection of scan rates from .1 mV/SEC to 500 mV/SEC. Use the SCAN RATE switch to adjust the scan rate, then select other scanning parameters and options as necessary for the measurement at hand, and use the RUN and CELL ON switches to apply the excitation waveform to the cell.

3.8D CONTROL I LINEAR SCANNING

The instrument's output in this mode is a current ramp, where the initial current is the product of the INITIAL POTENTIAL setting, used here as a dimensionless multiplier, and the CURRENT RANGE setting. For an initial current of 1 mA, for example, set the INITIAL POTENTIAL control to +1.000 and select a CURRENT RANGE of 1 mA. Similarly, for a final current of −1 mA, it is necessary to multiply the CURRENT RANGE setting (1 mA) by a FINAL POTENTIAL setting of −1.000.

In CONTROL I LINEAR SCANNING, the CURRENT RANGE setting, the INITIAL POTENTIAL selection, and SCAN RATE setting all interact to determine the $\Delta i/\Delta t$. Use the following formula to select a ramp with characteristics suitable for the measurement at hand:

$$\Delta i/\Delta t = (\text{SCAN RATE/INITIAL POTENTIAL}) \times \text{CURRENT RANGE}$$

The CURRENT RANGE button selects a value in units of amperes, while the SCAN RATE switch is calibrated in units of mV/SEC and the INITIAL POTENTIAL parameter is in units of volts. The units of volts factor out of the formula, leaving an expression in units of amperes/sec, as is appropriate for the parameter. For example, if the INITIAL POTENTIAL is +1.000 (volts) and the SCAN RATE switch is 500 mV/SEC, the $\Delta i/\Delta t$ is 500 (mV/SEC)/1.000 (V), or 0.5/SEC, times the CURRENT RANGE. If the CURRENT RANGE were 1 µA, the $\Delta i/\Delta t$ would be 500 nA/SEC. If the CURRENT RANGE were

10 µA, the $\Delta i/\Delta t$ would be 5 µA/SEC, etc.

3.9 CONTROL I, CONSTANT I SCANNING

This mode requires that CONTROL I *(in)* and CONSTANT I *(in)* be selected. The output of this mode is a constant current whose polarity depends upon the cell voltage (working electrode with respect to reference electrode) and the INITIAL POTENTIAL and FINAL POTENTIAL settings. This mode is especially suited for controlling charge-discharge cycles of batteries and cells.

The polarity of the current first applied to the cell when RUN is pressed will either charge or discharge the cell as necessary to take the cell voltage from the INITIAL POTENTIAL level to the FINAL POTENTIAL level. The CURRENT RANGE and SCAN RATE controls work together to determine the magnitude of this current. Outlined in black on the right-hand side of the SCAN RATE switch is a group of numbers ranging from 10 to 200. These numbers correspond to a legend below the switch, also outlined in black, CURRENT % OF FULL SCALE. The scan rate selected determines the percentage of the full-scale CURRENT RANGE selection applied to the cell. For example, if the CURRENT RANGE is 10 µA and the SCAN RATE is 50%, the constant-current output of the instrument is 50% of 10 µA, or 5 µA.

Assume that the working electrode is connected to the positive terminal of a 9 V secondary (rechargable) battery and that the counter and reference electrodes are connected to the negative terminal. Assume further that the instrument's controls are set as follows:

CELL: OFF
$E_{INITIAL}$ *(released)*
HOLD: *(released*
MULTIPLE *(released)*
I CONSTANT I *(IN)*
MODE: CONTROL I *(in)*
INITIAL POTENTIAL: +8.000
FINAL POTENTIAL: +5.000
CURRENT RANGE: 1 mA
SCAN RATE: 20 % OF FULL SCALE
IR COMPENSATION: OFF

As soon as the CELL is switched on and RUN is pressed, the instrument will attempt to discharge the battery at a rate of 20% of 1 mA or 200 µA. This discharge will continue until the battery voltage reaches the FINAL POTENTIAL of +5.000 V. Since MULTIPLE scans are selected in this example, the current will again reverse polarity and charge the battery at a rate of 200 µA until its voltage reaches the INITIAL POTENTIAL setting of 8.000 V. The cycle will continue until the operator intervenes.

The discussion of the RAMP OUT, POTENTIAL MONITOR, and CURRENT MONITOR outputs in the Subsection 3.8 also applies here.

3.10 STEP FUNCTION RESPONSE

If a step function is applied to the EXT IN input, one might expect to see a faithful replica of the applied function at the CURRENT and POTENTIAL MONITOR outputs. This is not always the case. Sometimes, overshoot will be observed on the leading edge of the observed waveform, as shown in Figure III-2. The source of the overshoot is the time required for the cell current to charge the stray shunt capacitance of the cell cable.

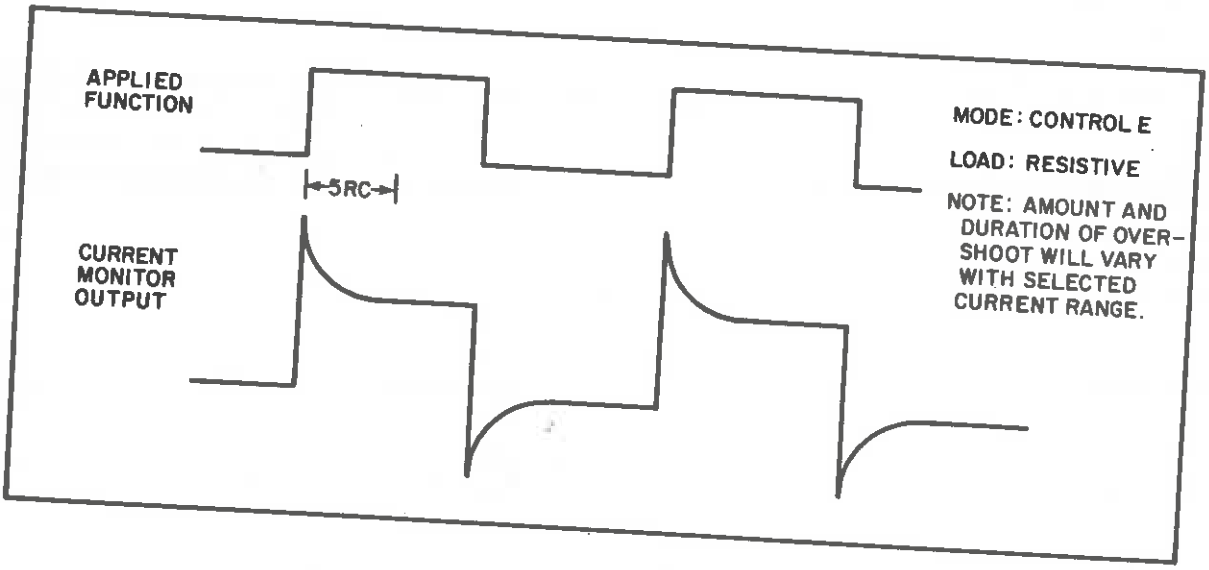

Figure III-2. STEP-FUNCTION OVERSHOOT

Although the observed overshoot will behave as illustrated with a resistive cell only, the effect may have to be considered when working with a real cell. Usually, the distortion caused by the cell's capacitance will dominate that due to charging of the cable capacitance. Nevertheless, the cable charging effect could be noticeable in certain applications.

Although the cable is guarded to minimize capacitance effects, typically 100 pF of stray capacitance remains. This capacitance, together with the range resistance across which the current signal is developed, forms an RC time constant. The effect of the time constant is to cause the current to be momentarily higher than the correct step level. As shown in the figure, this current asymptotically decays to the feedback-determined level in five time constants. The actual time constant varies with the selected current range since the value of "R" is different for each range as shown in Table III-2. The table also lists 5RC, the time required for the overshoot to decay, versus range so that the duration of the overshoot can be easily determined.

CURRENT RANGE	RESISTANCE	CAPACITANCE	5RC
1 A	0.5 Ω	100 pF	250 ps
100 mA	5.0 Ω	100 pF	2.5 ns
10 mA	50 Ω	100 pF	25 ns
1 mA	500 Ω	100 pF	250 ns
100 μA	5 kΩ	100 pF	2.5 μs
10 μA	50 kΩ	100 pF	25 μs
1 μA	500 kΩ	100 pF	250 μs

Table III-2. TYPICAL OVERSHOOT DURATION AS A FUNCTION OF CURRENT RANGE

As can be seen from the table, the overshoot spike will not be seen on the high current ranges where 5RC is less than the potentiostat rise time of ≈ 7 μs. The effect should only be appreciable on the 10 μA and 1 μA ranges.

It is important to understand that the observed effect is different for each mode. In CONTROL E operation, the signal observed at the POTENTIAL MONITOR connector will not show the aforementioned overshoot. That at the CURRENT MONITOR connector will. The situation is reversed in CONTROL I operation. The overshoot is observed at the POTENTIAL MONITOR connector, but not at the CURRENT MONITOR connector.

Applications could arise in which the user will wish to measure the current step-function response directly at the working electrode. Tektronix Inc. manufactures clip-on current probes designed for use with an oscilloscope. These probes are suitable for direct monitoring of the current waveform. The probe simply clips to the working electrode lead at the end of the cell cable, and the signal is observed at the associated oscilloscope.

3.11 RACK MOUNTING

Before the Model 362 can be rack mounted, it must be fitted with two mounting ears. These simply bolt to each side of the instrument at the front. The ears and mounting screws are available from EG&G PARC. The part numbers for the two ears are 1415-0780 and 1415-0781 respectively. The four required #8 screws are designated as part number 2811-0261.