

MODEL 362 SCANNING POTENTIOSTAT

OPERATING AND SERVICE MANUAL

 **EG&G** *PRINCETON APPLIED RESEARCH*

SHOULD YOUR EQUIPMENT REQUIRE SERVICE

- (A) Contact the factory (609-452-2111) or your local factory representative to discuss the problem. In many cases it will be possible to expedite servicing by localizing the problem to a particular plug-in circuit board.
- (B) If it is necessary to send any equipment back to the factory, we need the following information:
- (1) Model number and serial number.
 - (2) Your name (instrument user).
 - (3) Your address.
 - (4) Address to which instrument should be returned.
 - (5) 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 to
- (D) Customers outside of U.S.A.: To avoid delay in customs-clearance of equipment being returned, please contact the factory or the nearest factory distributor for complete shipping information.
- (E) Address correspondence to

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SECTION I CHARACTERISTICS

1.1 SAFETY NOTICE (READ BEFORE OPERATING INSTRUMENT)

1.1A 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 to insure safe operation and to retain the apparatus in a safe condition. The described apparatus has been designed for indoor use.

1.1B INSPECTION

Newly received apparatus should be inspected for shipping damage. If any is noted, notify EG&G PARC and file a claim with the carrier. Be sure to save the shipping container for 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 PERSONNEL. DAMAGED APPARATUS WAITING FOR SAFETY VERIFICATION SHOULD BE TAGGED TO INDICATE TO A POTENTIAL USER THAT IT MAY BE UNSAFE AND THAT IT SHOULD NOT BE OPERATED.

1.1C SAFETY MECHANISM

As defined in IEC Publication 348 (*Safety Requirements for Electronic Measuring Apparatus*), this 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 shall be connected to a protective conductor. The connection is made via the earth ground prong of the power cord plug. The power cord plug shall only be inserted in a socket outlet provided with the required earth ground contact. The protective action must not be negated by the use of an extension cord or adapter plug without a protective conductor.

WARNING!

ANY INTERRUPTION OF THE PROTECTIVE CONDUCTOR INSIDE OR OUTSIDE THE APPARATUS OR DISCONNECTION OF THE PROTECTIVE EARTH TERMINAL MAY MAKE THE APPARATUS DANGEROUS. INTENTIONAL INTERRUPTION IS PROHIBITED.

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

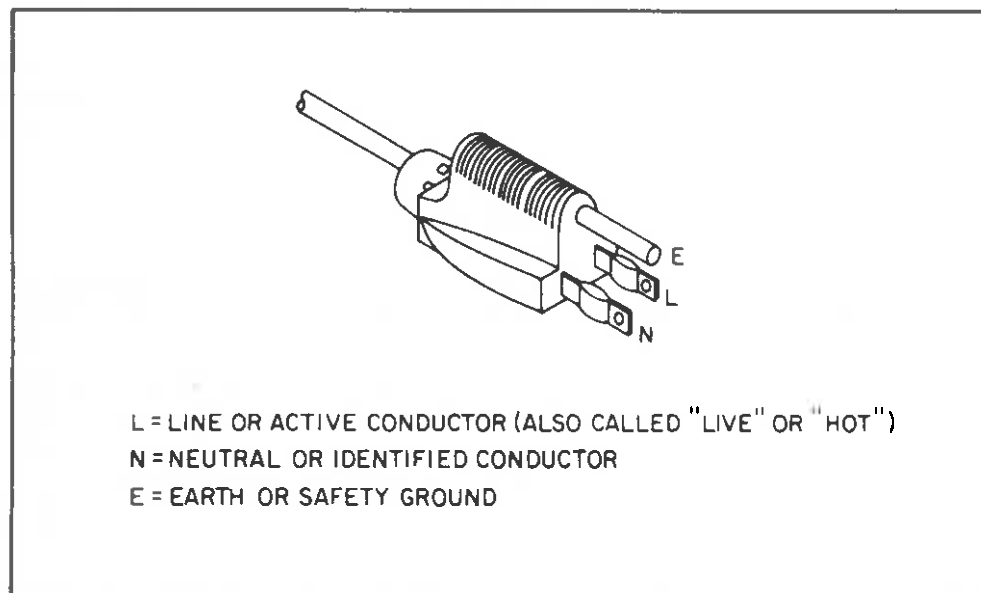


Figure I-1. POWER CORD PLUG WITH POLARITY INDICATION

1.1D DEFECTS

Whenever it is likely that the protection provided by the connection to earth ground has been impaired, the apparatus should be made inoperative and secured against unintended operation. The protection is likely to be impaired if, for example, the apparatus

- (1) shows visible damage,
- (2) fails to perform the intended measurements,
- (3) has been subjected to prolonged storage under unfavorable conditions, or
- (4) has been subjected to severe transport stresses.

Such apparatus should not be used until its safety has been verified by qualified service personnel.

1.1E CABLE LEADS

The Model 362 has a compliance of ± 30 V at 1 A. Thus potentials as high as ± 30 V can be present at the counter electrode lead of the cell cable. Although 30 V is not generally regarded as being a dangerous potential, THE HIGH CURRENT CAPABILITY REQUIRES THAT REASONABLE PRECAUTIONS BE TAKEN IN HANDLING THESE LEADS. The CELL switch should always be set to OFF when making the cell connections. it should similarly be set to OFF when the cable leads are being examined or disconnected.

WARNING!

IF IT IS NECESSARY TO REPLACE THE POWER CORD OR THE POWER 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 PERSONAL INJURY OR DEATH, MIGHT EXIST.

1.1F POWER VOLTAGE SELECTION

Before plugging in the power cord, make sure that the equipment is set to the voltage of the AC power supply. A detailed discussion of how to check and, if necessary, change the power voltage setting follows.

The Model 362 can operate from any of four different power voltage ranges, 90-110 V, 110-130 V, 180-220 V, and 220-260 V, 50-60 Hz. Change from one voltage range to another is made by repositioning a plug-in circuit card internal to 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 the 220-260 V range. If this is the case, they are shipped configured for operation from a voltage in the higher 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. Observing the instrument from the rear, note the clear-plastic "door" immediately adjacent to the power cord connector (Figure I-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 board. The selector board is located at the lower edge of the fuse compartment. A number printed on the upper surface of the selection board is visible without removing the board. 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 board, but only one is visible at a time. In other words, the board can be inserted in any of four different positions, and a different number can be read in each. Table I-1 indicates the actual line voltage range for each number. If the number showing is incorrect for the prevailing line voltage, the board will have to be repositioned, as described in the following paragraphs.

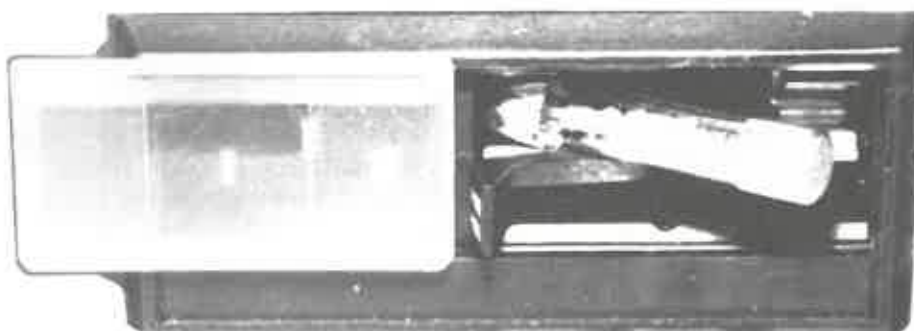


Figure I-2. LINE CORD/FUSE ASSEMBLY

The first step is to remove the fuse. When the lever labeled FUSE PULL is rotated out and toward the left, the fuse will lift so that it can be easily removed. At the front center of the circuit board is a small hole that serves as a convenient pry point. A small screwdriver or other tool 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 I-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 pushed all the way in. Then check the fuse rating. For operation from a nominal line voltage of 120 V, use a slow-blow fuse rated at 1 A (LITTELFUSE 3AG) or equivalent). 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 and of the specified type are used for replacement. The use of makeshift fuses and the short-circuiting of fuse holders are prohibited.

SELECTOR BOARD
NUMBER EXPOSED

OPERATING
VOLTAGE RANGE

100 -----	90-110 V
120 -----	110-130 V
220 -----	180-220 V
240 -----	220-260 V

Table I-1. VOLTAGE-SELECTION BOARD POSITION
AS A FUNCTION OF LINE VOLTAGE

1.1G VENTILATION

The Model 362 does not incorporate forced-air ventilation. With a power consumption of only 75 watts maximum, this instrument can be operated on any laboratory bench. Alternatively, it can be rack mounted, if desired. The only requirement is that the ambient temperature be restricted to a range of 15° to 45°C. If the temperature in the rack is near the high end of this range, forced air cooling of the rear-panel heat sink may be desirable, particularly if the Model 362 is to be operated for long periods at high current levels. Always leave adequate space behind the instrument for air to circulate freely. Six inches (15 cm) should be sufficient to prevent overheating.

1.2 LINE VOLTAGE AND EQUIPMENT SAFETY

As previously discussed, the Model 362 can be operated from any of four different voltage ranges as given in Table I-1. The procedure for adapting the instrument from one range to another is provided in Subsection 1.1F.

CAUTION!

THE APPARATUS DESCRIBED IN THIS MANUAL MAY BE
DAMAGED IF THE VOLTAGE APPLIED IS ABOVE THE
UPPER LIMIT OF THE SELECTED RANGE.

1.3 INTRODUCTION

The Model 362 Scanning Potentiostat is a $\pm 1A$, $\pm 30 V$ potentiostat and galvanostat, as well as a versatile ramp generator capable of applying a ramping voltage or current to a electrochemical cell. The instrument can also charge and discharge a cell or battery at a constant current between pre-set INITIAL POTENTIAL and FINAL POTENTIAL levels.

All indicators, controls, and input/output connectors are located on the instrument's front-panel. Indicators include an analog meter for displaying the polarity and instantaneous magnitude of the cell's voltage or current. There are also two OVERLOAD lamps. One, the CURRENT OVERLOAD lamp, indicates that the cell current is more than twice the meter's full-scale range. The other, the CONTROL AMPLIFIER OVERLOAD lamp, turns on when the instrument's power amplifier is out of its linear operating region. Other indicators include an illuminated power switch and an CELL ON-OFF switch with a highlighted ON position. Controls include a pushbutton MODE switch for selecting CONTROL E or CONTROL I OPERATION, a bank of seven pushbutton CURRENT RANGE selectors, and a METER pushbutton that alternates the meter display between voltage and current readings. Two 4-digit thumbwheel switches establish voltage or current parameters for the instrument's static and scanning modes.

For operation in the CONTROL E MODE, the front panel also includes IR COMPENSATION controls, which compensate for the cell resistance between the reference electrode and the working electrode. (A feedback loop automatically adjusts for cell resistance between the counter electrode and the working electrode.) These controls apply positive feedback to the instrument's control amplifier.

When the instrument is in one of its flexible scanning modes, a group of front-panel controls and selectors interact to set the scanning parameters and select among a broad range of options. A bank of pushbutton switches allows the operator to initiate scans, select single or multiple scans, determine the endpoints for single scans, and reverse the direction of a scan that is underway. Scan control switches also allow the operator to halt scanning and either to freeze the output at its instantaneous level or to halt scanning and restore the initial level. The INITIAL POTENTIAL and FINAL POTENTIAL thumbwheel switches establish the turn-around points for the ramp function in CONTROL E scanning as well as the limits for the cell or battery potential in the CONTROL I, CONSTANT I scanning mode. In CONTROL I scanning, the INITIAL POTENTIAL setting serves as dimensionless multiplier of the CURRENT RANGE setting to establish the initial cell current.

A rotary SCAN RATE switch determines $\Delta v/\Delta t$ directly for CONTROL E scanning. This switch interacts with the INITIAL POTENTIAL and CURRENT RANGE settings to determine $\Delta i/\Delta t$ in CONTROL I scanning. In CONTROL I, CONSTANT I scanning, the SCAN RATE switch sets the constant current as a percentage of the full-scale CURRENT RANGE setting.

Any voltage applied to the EXT IN BNC connector is summed with the INITIAL POTENTIAL setting and presented to the input of the control amplifier. Thus a voltage generated by an external device or system can set or alter the cell's current or voltage. The instrument's provisions for signal output consist of three sets of binding posts, each set designed for straightforward connection to external apparatus such as X-Y or strip-chart recorders. The set labeled CURRENT MONITOR presents a 0 to 1 V signal proportional to the full-scale current. The POTENTIAL MONITOR connectors carry a signal in the range of $\pm 9.999V$ and equal to the potential of the reference electrode with respect to the working electrode. The RAMP OUT connectors output a signal equal in magnitude (though opposite in polarity) to the ramping component of the voltage applied to the cell. This signal is also defined as $-E_{\text{working}}$ vs. E_{initial} . These signals are provided for connection to peripheral monitoring instrumentation or for use as feedback signals in complex control systems.

The Model 362 includes a built-in, unity-gain electrometer to buffer the reference electrode.

1.4 SPECIFICATIONS

Unless otherwise noted, all specifications assume a potential measured at the working electrode with respect to the reference electrode, a load resistance of 10 k Ω between the working and reference electrodes, and an ambient temperature of 25°C.

STATIC FUNCTIONS

CONTROL E MODE

APPLIED POTENTIAL RANGE: $\pm 9.999 V$ TO $\pm 9.999 V$.

APPLIED POTENTIAL RESOLUTION: 1 mV steps.

APPLIED POTENTIAL ACCURACY: $\pm 3 \text{ mV} \pm 0.4\%$ of INITIAL POTENTIAL SETTING.

CONTROL AMPLIFIER
OUTPUT VOLTAGE

35 Ω

$> \pm 30 V$.

MAXIMUM OUTPUT CURRENT

1 Ω resistive load

$> 1 A$.

RISE TIME

10% to 90% with 1 V
step function applied
to EXT IN BNC:

10 k Ω resistive load: <10 μ s.
1 Ω resistive load: <15 μ s.

DRIFT WITH TIME: <200 μ V/week.

DRIFT WITH TEMPERATURE: <30 μ V/ $^{\circ}$ C + 200 ppm of INITIAL
POTENTIAL setting per $^{\circ}$ C.

NOISE AND RIPPLE
1 Hz to 3 kHz: <200 μ V rms.

LINE VOLTAGE SENSITIVITY
 $\pm$ 10% change in line: <200 μ V.

STABILITY WITH
CAPACITIVE LOADS: Stable with Reference
Electrode Impedance <30 k Ω .

SLEW RATE >25 V/ μ s.

CONTROL I MODE

ACCURACY: Better than $\pm$ 0.4% of full-
scale CURRENT RANGE setting.

DRIFT WITH TIME: <0.02% of full-scale CURRENT
RANGE/week.

DRIFT WITH TEMPERATURE: <0.01% of full-scale/ $^{\circ}$ C.

NOISE AND RIPPLE: <200 μ V rms.

RISE TIME
10% to 90% with 100
 μ A peak-to-peak step
function applied to
EXT IN BNC: <10 μ s.

CURRENT MEASUREMENT RISE TIME: 2 μ s.

ELECTROMETER

INPUT VOLTAGE
Reference electrode
with respect to
working electrode: $\pm$ 10 V.

INPUT CURRENT
At 25 $^{\circ}$ C: 100 pA typical, 2 nA maximum.

*h = 19 cm
length = 64.5 cm
width = 39.0 cm*

panels 8.1 kg

INPUT CAPACITANCE DC to 200 kHz:	<20 pF.
INPUT RESISTANCE:	$2 \times 10^{10} \Omega$.
FREQUENCY RESPONSE -3 dB with 1 Ω source:	>1 MHz.
DRIFT WITH TIME:	<200 μ V/week.
DRIFT WITH TEMPERATURE:	<30 μ V/ $^{\circ}$ C.

SCANNING FUNCTIONS

CONTROL E, LINEAR SCANNING

SCAN RANGE:	± 9.999 V to ± 9.999 V.
LIMIT RESOLUTION:	1 mV steps.
LIMIT ACCURACY:	± 3 mV $\pm$ 0.4% of INITIAL POTENTIAL setting.
SCAN RATE RANGE:	0.1 mV/s to 500 mV/s selected with front panel rotary switch.
SCAN RATE MULTIPLIER SWITCH:	Internal X10 slide switch.
SCAN RATE RESOLUTION:	Selectable in multiples of 1, 2, and 5.
SCAN RATE ACCURACY:	± 0.01 mV/s $\pm$ 0.5% SCAN RATE setting.
TURN-AROUND ACCURACY (The point at which the slope of the scan- ning waveform changes polarity):	POTENTIAL setting ± 2 mV $\pm$ 0.002 X SCAN RATE setting.

CONTROL I, LINEAR SCANNING

APPLIED CURRENT RANGE:	100 nA to >1 A.
SCAN RATE RANGE:	1 nA/s to 500 mA/s.
SCAN RATE RESOLUTION:	Selectable in multiples of 1, 2, and 5.
SCAN RATE ACCURACY	$\pm 1 \times 10^{-5} \times \text{CURRENT RANGE} \pm 0.5\% \text{ OF SCAN RATE.}$
SCAN RATE MULTIPLIER SWITCH:	Internal X10 slide switch.
TURN-AROUND ACCURACY:	$\pm 2 \times 10^3 \times \text{CURRENT RANGE} \pm 0.02\% \text{ OF SCAN RATE.}$

CONTROL I, CONSTANT I LINEAR SCANNING

APPLIED CURRENT RANGE:	100 nA to 1 A.
ACCURACY:	$\pm 0.4\% \text{ OF CURRENT RANGE} \pm 0.5\% \text{ resolution setting.}$
RESOLUTION:	10% to 200% of full-scale CURRENT RANGE setting in multiples of 1, 2, and 5. (10% to 100% of 1 A range.)
INITIAL AND FINAL THRESHOLDS:	$\pm 9.999 \text{ V to } \pm 9.999 \text{ V.}$
THRESHOLD RESOLUTION:	1 mV.
TURN-AROUND ACCURACY:	POTENTIAL setting $\pm 2 \text{ mV} \pm 0.002 \times \text{SCAN RATE setting.}$

1.5 DESCRIPTION

1.5A CONTROL E MODE

Figure I-3 is a simplified block diagram of the Model 362 operated in the static Control E mode. As is shown in the figure, the control loop is simple. A voltage, E_A , is applied to the input. This voltage supplies current through a resistor R (all resistors of value R in the diagram are 10 k Ω) to the summing junction of the control amplifier. To satisfy the loop requirements, it is necessary that the summing junction of the control amplifier be at 0 V. As shown, there are two feedback paths, one from the reference electrode, the other from the working electrode, to the summing junction. They work together to

establish the summing junction at the required 0 V level. Because E_A is applied to the inverting input of the control amplifier, the output of the amplifier is driven in the opposite direction. The amplifier drives the counter electrode. Note that the working electrode is returned to ground through R_L , a resistance determined by the selected current range. This resistance does not affect the cell current, but does determine the working electrode potential. The reference electrode will be at some intermediate potential. The objective in Control E operation is to establish the working electrode at E_A with respect to the reference electrode. Because of the drop across R_L , the working electrode is at a potential of iR_L volts, and the potential of the working electrode with respect to the reference electrode is simply $iR_L - E_r$, where E_r is the potential of the reference electrode with respect to ground.

Consider the two feedback paths. E_r is fed back directly to the summing junction of the power amplifier through a resistor of value R (U2 is a non-inverting unity-gain electrometer amplifier).

The working electrode potential, on the other hand, is inverted by U1 to become $-R_L$ before being applied to the summing junction. With this arrangement, for the loop to be satisfied, $E_A + E_r - iR_L = 0$, or $E_r = iR_L - E_A$.

Recall that the potential of the working electrode with respect to the reference electrode is $iR_L - E_r$. In this expression, $iR_L - E_A$ can be substituted for E_r , giving E_w (potential of working electrode with respect to the reference electrode) = E_A , the desired relationship. In other words, the loop always works to make the working electrode potential (with respect to the reference electrode, not with respect to ground) the same as the applied potential.

The remaining circuits shown develop the POTENTIAL and CURRENT MONITOR Outputs. The POTENTIAL MONITOR output is simply the reference electrode potential with respect to the working electrode potential (each measured with respect to ground), and thus will be the same as E_A but of opposite polarity. The CURRENT MONITOR output is $-2R_L$ (the factor of two gain is provided by amplifier U8). The values of R_L and the gain are such as to provide a CURRENT MONITOR output voltage of 1 V if the cell current is full scale. As indicated, the panel meter connects to either the POTENTIAL MONITOR Output (meter polarity reversed) or to the CURRENT MONITOR Output (meter polarity same as Output polarity).

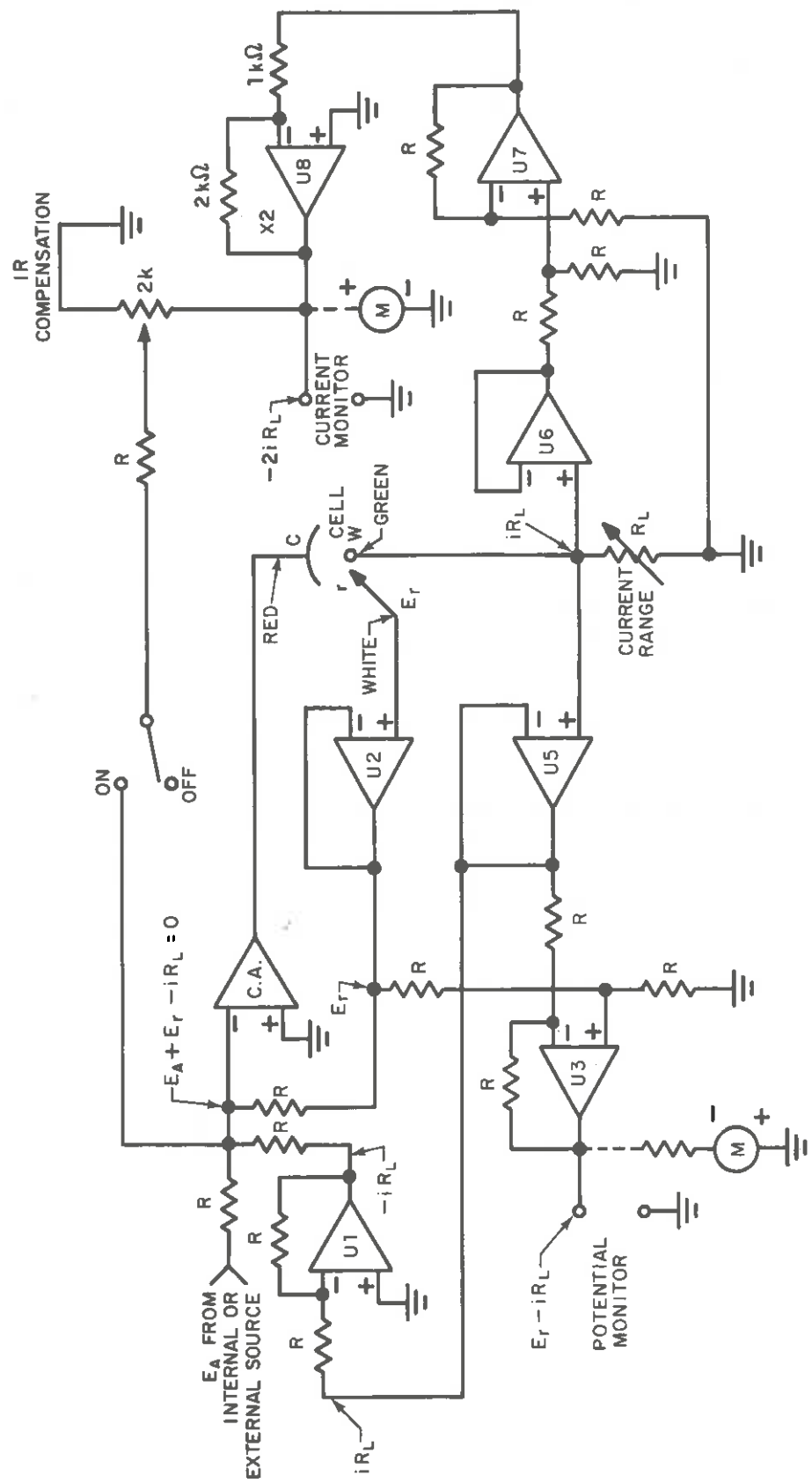


Figure I-3. CONTROL E MODE

1.5B CONTROL I MODE

Figure I-4 is a simplified block diagram of the Model 362 operated in the static Control I mode. As shown, the control amplifier output drives the counter electrode. The polarity of that drive is inverted with respect to the input, E_A . As in Control E operation, the working electrode potential is iR_L . Since this voltage is the only one used to develop the feedback signal (the reference electrode potential is not in the control loop), the cell current is controlled independent of the reference electrode potential.

U6 is a non-inverting buffer that drives differential amplifier U7. The arrangement used prevents errors due to the voltage drop across the resistance of the cable's working electrode lead. The output of U7, also iR_L , is applied to the summing junction of the power amplifier through a resistor of value $R/2$. Because this resistor is $1/2$ the resistance of the input resistance seen by E_A , the loop equation is $E_A + 2iR_L = 0$, or $i = -E_A/2R_L$. In other words, the cell current is of opposite polarity to E_A , and its magnitude is $E_A/2R_L$. The value of R_L is determined by the selected current range such that with an E_A of 1 V, i will equal one full scale on the selected current range, and yield +1 V out at the CURRENT MONITOR output.

The POTENTIAL MONITOR output is the same as in Control E operation, that is, the reference electrode potential with respect to the working electrode is provided at the POTENTIAL MONITOR output. If desired, the reference electrode can be omitted in CONTROL I operation, in which case the POTENTIAL MONITOR output voltage is indeterminate. Also, the panel meter connections are the same as in the CONTROL E mode.



1.5C LINEAR SCANNING AND CYCLIC MODES

Figure I-5 is a simplified block diagram showing the ramp generator and control logic that produces the instrument's scanning and cyclic functions. A central component of this functional diagram is U3009, an operational amplifier whose output is either a ramp function or a DC level. In the scanning and cyclic modes, this output becomes E_A (see Figures I-3 and I-4), the input to the the CONTROL AMPLIFIER, shown in these figures as an operational amplifier labeled C.A.

In the LINEAR SCAN mode, the front panel LINEAR SCAN-CONSTANT I switch opens K3004 and closes K3003 to create a feedback path for U3009 through the 40 μ F INTEGRATOR capacitor. As a result, operational amplifier U3009 BECOMES an integrator, and its output is a linear ramp whose slope is determined by the magnitude and polarity of the output of U4002, applied through the closed contacts of K3002. (The HOLD button and the LOGIC CIRCUIT can open this relay to halt ramping.) If the instrument is in its CONTROL I, CONSTANT I MODE, K3003 opens and K3004 closes, shunting the integrator capacitor, and creating a feedback path through the CONST I CAL resistor. In this instance, U3009 is simply an inverting amplifier, and its output is a DC level that ultimately results in a constant-current output from the CONTROL AMPLIFIER.

The SCAN RATE switch, which varies the resistance between +15 V and the input of U4002, determines the magnitude of U4002's output. Relay K4001 determines its polarity. When K4001 opens, U4002 serves as a unity-gain, non-inverting amplifier. When the relay closes, U4002 serves as a unity gain, inverting amplifier. Whether this relay is open or closed is determined by the LOGIC CIRCUIT, which decodes not only the front-panel scan-control switches, but also the outputs of U3002, U3008, and U3005, arranged as a system of comparators.

If the RUN- $E_{INITIAL}$ switch is in the RUN position, K3001 opens, and the output from U3009 is applied to the CONTROL AMPLIFIER. If this switch is in the $E_{INITIAL}$ position, and the LINEAR SCAN-CONSTANT I switch is in the LINEAR SCAN position, the INITIAL POTENTIAL is applied to the input of the CONTROL AMPLIFIER through K3001. If this switch is in the CONSTANT I position while RUN is engaged, K3003 closes, converting U3009 from an integrator to an amplifier, and the POTENTIAL MONITOR output is applied to the comparator circuit. U3006 is simply an inverting buffer that drives the RAMP OUT output with a voltage that is equal in magnitude and opposite in polarity to the ramping voltage applied to the control amplifier. Note that $E_{initial}$ is nulled out of the RAMP OUT signal, while both $E_{initial}$ and the ramping voltage are components of the POTENTIAL MONITOR signal.



Figure 1-5. SCANNING MODES