MODEL 173 POTENTIOSTAT/ GALVANOSTAT

SEE SAFETY NOTICE PRECEDING SECTION I BEFORE OPERATING INSTRUMENT



FINCETON APPLIED RESEARCH

MODEL 173 POTENTIOSTAT/ GALVANOSTAT

OPERATING AND SERVICE MANUAL



WARNING

When operating in the CONTROL E or CONTROL I mode, voltages as high as 100 V with a current capability of 1 A may be present at the external cell cable alligator clips when the Selector switch is in the EXT. CELL position. To minimize the possibility of dangerous, possibly lethal electrical shock, operators are warned not to touch these exposed clips, either to connect to a cell, or to disconnect from it, unless the Selector switch is first placed in the OFF position.

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

EG&G PRINCETON APPLIED RESEARCH 7 Roszel Road (Off Alexander Road, East of Route 1) Princeton, New Jersey

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

EG&G PRINCETON APPLIED RESEARCH P. O. Box 2565 Princeton, NJ 08540

Phone: 609/452-2111 TELEX: 84 3409

WARRANTY

EG&G PRINCETON APPLIED RESEARCH warrants each instrument of its manufacture to be free from 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, freight prepaid, to our factory within ONE year of delivery to the original purchaser, provided prior authorization for such return has been given by our authorized representative.

and the same

This Warranty shall not apply to any instrument which our inspection shall disclose to our satisfaction, has become defective or unworkable due to abuse, mishandling, misuse, accident, alteration, negligence, improper installation or other causes beyond our control. Instruments manufactured by others, and included in or supplied with our equipment, are not covered by this Warranty but carry the original manufacturer's warranty which is extended to our customers and may be more restrictive. Certain subassemblies, accessories or components may be specifically excluded from this Warranty, in which case such exclusions are listed in the Instruction Manual supplied with each instrument.

We reserve the right to make changes in design at any time without incurring any obligation to install same on units previously purchased.

THERE ARE NO WARRANTIES WHICH EXTEND BEYOND THE DESCRIPTION HEREIN. THIS WARRANTY IS IN LIEU OF, AND EXCLUDES ANY AND ALL OTHER WARRANTIES OR REPRESENTATIONS, EXPRESSED, IMPLIED OR STATUTORY, INCLUDING MERCHANTABILITY AND FITNESS. AS WELL AS ANY AND ALL OTHER OBLIGATIONS OR LIABILITIES OF EG&G PRINCETON APPLIED RESEARCH, INCLUDING, BUT NOT LIMITED TO, SPECIAL OR CONSEQUENTIAL DAMAGES. NO PERSON, FIRM OR CORPORATION IS AUTHORIZED TO ASSUME FOR EG&G PRINCETON APPLIED RESEARCH ANY ADDITIONAL OBLIGATION OR LIABILITY NOT EXPRESSLY PROVIDED FOR HEREIN EXCEPT IN WRITING DULY EXECUTED BY AN OFFICER OF EG&G PRINCETON APPLIED RESEARCH.

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SAFETY NOTICE (Read before operating instrument)

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.

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 SERVICE 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.

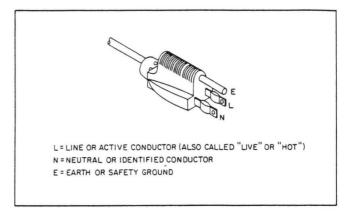
SAFETY MECHANISM

As defined in IEF Publication 348 (Safety Requirements for Electronic Measuring Apparatus), this is Class I apparatus, that is, this apparatus 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 protective 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 reguired 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.

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 power 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.



POWER CORD PLUG WITH POLARITY INDICATIONS

WARNING!

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

POWER VOLTAGE SELECTION

Before plugging in the power cord, make sure that the equipment is set to the voltage of the ac power supply. For safety, this check should only be made with the instrument disconnected from any source of power. The check is made as follows. NOTE: FOR SAFETY, THIS PROCEDURE SHOULD ONLY BE PERFORMED BY QUALIFIED SERVICE PERSONNEL.

- (1) Remove the screws securing the top cover and slide it back to where it is free of the instrument.
- (2) Looking down into the Model 173, note the line voltage selector switch mounted on the chassis directly in front of the power transformer. For operation from a voltage in the range of 105-125 V ac, 50-60 Hz, "115" should be visible through the switch window. For operation from a voltage in the range of 210-250 V ac, 50-60 Hz, "230" should show.
- (3) If the voltage selector is in the wrong position for the prevailing line voltage, change the switch setting as necessary, using a small screwdriver as the actuating tool.

(4) When finished, return the top cover to the instrument and secure it.

CAUTION!

THE APPARATUS DESCRIBED IN THIS MANUAL MAY BE DAMAGED IF THE LINE VOLTAGE SELECTOR SWITCH IS INCORRECTLY SET.

FUSES

The ac power fuse is located at the rear panel. For operation from a line voltage of nominally 115 V, use a slow-blow 5 A fuse with a voltage rating of 125 V or higher. For operation from a line voltage of nominally 230 V, use a slow-blow 3 A fuse with a voltage rating of 250 V or higher.

In addition, there are six internal fast-blow fuses that protect the dc supplies. The current rating of these fuses is indicated in Figure V-1 on page V-1. In each case, use a fast-blow fuse with a voltage rating equal to, or higher than, the voltage indicated in Figure V-1.

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

WARNING!

TO AVOID THE POSSIBILITY OF A SAFETY HAZARD FROM ELECTRICAL SHOCK WHICH COULD RESULT IN PERSONAL INJURY OR DEATH, DISCONNECT THE POWER CORD BEFORE REMOVING OR INSTALLING A FUSE. FUSES SHOULD ONLY BE CHANGED BY QUALIFIED SERVICE PERSONNEL.

CELL CONNECTIONS

When operating in the CONTROL E or CONTROL I mode, voltages as high as 100 V with a current capability of 1 A may be present at the external cell cable alligator clips when the Selector switch

is in the EXT. CELL position. To minimize the possibility of dangerous, possibly lethal electrical shock, operators are warned not to touch these exposed clips, either to connect to a cell, or to disconnect from one, unless the Selector switch is first placed in the OFF position.

VENTILATION

The Model 173 uses forced-air ventilation to maintain a safe internal operating temperature. For this reason, it is essential that there be adequate air circulation to the rear of the instrument. Practically speaking, this is readily accomplished by allowing at least 15 cm between the rear of the instrument and the nearest obstruction (back of bench, wall, etc.). If the unit is operated in an enclosed space, such as an equipment cabinet, it is important that there be good circulation between the cabinet and the rest of the lab. Some equipment cabinets incorporate forced air cooling to assure adequate ventilation.

DEFECTS AND ABNORMAL STRESSES

Whenever it is likely that the protection provided by the connection to earth ground has been impaired, the apparatus shall be made inoperative and secured against any 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.
- (4) Has been subjected to severe transport stresses.

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

SECTION I CHARACTERISTICS

1.1 INTRODUCTION

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The Model 173 Potentiostat/Galvanostat is a dualfunction electrochemical measuring instrument. Operated as a potentiostat, it holds the potential between two electrodes of an electrochemical cell constant, or it changes the voltage between two electrodes in a very specific, carefully controlled manner, despite large changes in the current demanded by the system. Operated as a galvanostat, it holds the current through a cell constant, or it changes the current through a cell in a controlled manner, despite large changes in the applied potential required to maintain current control.

The Model 173 offers complete flexibility in potential or current control for electrochemical applications. The instrument features a current capability of one ampere with compliance voltages as high as 100 V in either polarity, and a slew rate of 10 V per microsecond. It incorporates two independent built-in potential/current sources, each adjustable to any voltage in the range of ± 4.999 V, as well as complete logic and switching circuitry for controlling the sources from the front panel or by externally derived triggers. Two additional external potential/current programming signals may be added to those provided by the instrument, and a wide variety of triggering and switching waveforms may be employed to control the applied potential/current programs.

A Model 178 Electrometer Probe is supplied with every Model 173 so that the potential at a high-impedance point (Reference Electrode) can be monitored. By placing the probe at the end of a cable, the probe can be positioned very near the monitored point. As a result, stray capacitance loading is minimized with a subsequent optimization of loop-stability parameters.

Several different plug-in modules are available for use with the Model 173, allowing the system performance to be tailored to the specific requirements of each measurement application. Available modules include the Model 176 Current-to-Voltage Converter, the Model 179 Digital Coulometer, and the Model 376 Logarithmic Current Converter.

With its wide compliance-voltage capability, heavy current capacity, and flexible control features, together with the adaptability provided by the choice of different plug-in modules, the Model 173 should see application in most laboratories where electrochemical measurements are conducted.

1.2 MAIN-FRAME SPECIFICATIONS

POTENTIAL/CURRENT CONTROL

 INTERNAL: Two separate potential sources are provided, each adjustable from 0 V to

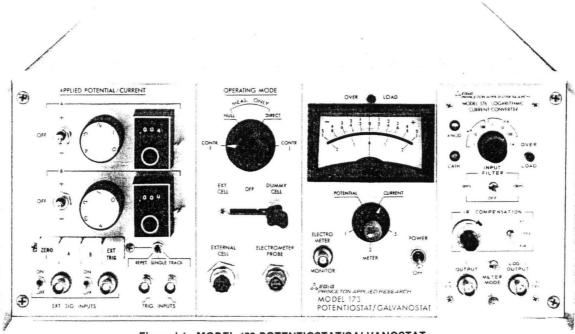


Figure I-1. MODEL 173 POTENTIOSTAT/GALVANOSTAT

 \pm 4.999 V. Setting resolution is \pm 1 mV and accuracy is 0.1% ±1 mV. In galvanostatic operation these controls operate in conjunction with the Range controls on the plug-in module to set the level at which the current is controlled.

The output of one potential source or the other controls the potentiostat/galvanostat as determined by front-panel pushbuttons. There is provision for switching from one potential source to the other by means of externally derived triggers.

(2) EXTERNAL: Two connectors are provided to allow externally derived control programs to be added to the output of either internal source. The control potential at any time is the sum of three potentials. They are (i) the output of one of the two internal sources, (ii) the potential applied to the first EXT. SIG. IN-PUT, and (iii) the potential applied to the second EXT. SIG. INPUT. The input impedance at these connectors is 10 k Ω . Associated toggle switches allow externally derived signals to be disconnected from the control circuits, if desired.

MODES

- (1) CONTROL E: Instrument functions as a potentiostat, allowing the Working Electrode of an electrochemical cell to be maintained at the programmed potential with respect to the Reference Electrode. This is done by applying a voltage to a third (Counter) electrode. The potential applied to the Counter Electrode is automatically servoed to whatever value is required (within the ± 100 V compliance capabilities of the instrument) to maintain the programmed potential relationship between the Reference and Working Electrodes. The mainframe can function in the CONTROL E mode by itself, although there is no way of measuring the cell current.
- (2) CONTROL I: Instrument functions as a galvanostat, maintaining the programmed current flow through an electrochemical cell. The voltage across the cell is automatically servoed (within the ± 100 V compliance capabilities) to the voltage required to maintain the desired current. CONTROL I operation is only possible if the unit is equipped with a plug-in module (M176, M179, M376).
- (3) MEASURE ONLY NULL: Voltage monitored by Model 178 Electrometer is read out by adjusting the Applied Potential/Current controls for "0" meter indication and reading the voltage from the setting of these controls. MEASURE ONLY NULL operation can be accomplished with the mainframe alone.

(4) MEASURE ONLY DIRECT: Voltage monitored by Model 178 Electrometer is displayed directly on the meter. MEASURE ONLY DIRECT operation can be accomplished with the mainframe alone.

MEASUREMENT CAPABILITIES

- (1) FRONT-PANEL ANALOG METER: There are two measurement modes, POTENTIAL and CURRENT, as selected by the associated Meter switch. In POTENTIAL, meter indicates potential sensed by the Model 178 Electrometer Probe. Full scale is 1, 2, or 5 V as selected by the Meter Range switch. In CURRENT, meter indicates programmed current relative to Current Range selected at plug-in module. Current measurements are only possible if plug-in module is installed.
- (2) FRONT-PANEL DIGITAL PANEL METER: Instruments equipped with Digital Panel Meter in place of analog meter are available. Meter is 31/2 digits with BCD output of monitored parameter provided at rear-panel connector. Range data (BCD) is provided by plug-in module (M176D, M179, M376). All BCD data conforms to the following specifications:

Logic "0" = $+ 0.2 \text{ V} \pm 0.2 \text{ V}$; 5 mA maximum sinking current Logic "1" = $+3.5 \text{ V} \pm 1.0 \text{ V}$; 0.1 mA maximum sourcing current

A table of pin assignments is provided in Section IV.

FRONT-PANEL CONNECTORS

- (1) EXTERNAL SIGNAL INPUTS: Two connectors are provided, each with an associated ON-OFF toggle switch. Externally derived signals applied to these connectors directly add to the output of whichever of the two internal sources is active. Input impedance of both connectors is 10 k Ω .
- (2) TRIGGER INPUTS: Two connectors are provided to allow externally derived trigger signals to determine which of the two internal sources is active. Associated toggle switch gives choice of three control modes. Signal requirements are:

Minimum Trigger Voltage: 3 V Minimum Trigger Risetime: 0.1 μs Maximum Trigger Risetime: 100 μ s

Maximum Trigger Rate: 200 kHz with 5 V pk-

pk square wave trigger

(3) EXTERNAL CELL: Cable terminated in three color-coded alligator clips connects here and to the cell. In connecting to the cell, red connects to the Counter Electrode, green to the

Working Electrode, and Black is ground (need not be connected in most situations).

- (4) ELECTROMETER PROBE: The Model 178 Electrometer Probe cable must be connected and the probe must monitor the Reference Electrode for CONTROL E operation. The probe cable need not be connected for CON-TROL I operation, although it can be if desired.
- (5) ELECTROMETER MONITOR: The potential monitored by the M178 Electrometer Probe is provided at this connector. The source impedance is 1000 ohms.

REAR-PANEL CONNECTORS

- (1) DIGITAL INTERFACE: In units equipped with digital panel meter, measured parameter is provided at this connector in BCD format. (See Section IV for pin assignments.)
- (2) TRIG. OUT A & B: Outputs suitable for triggering external monitoring instruments are provided at these two connectors. A + 1 V level shift takes place at this connector when internal source A becomes active (either by pushbutton or by external trigger). When internal source B becomes active (either by pushbutton or by external trigger), the level at the A TRIG. OUT connector returns to zero and that at the B TRIG. OUT connector goes to +1 V.
- (3) S/A OUT: The control amplifier output (voltage applied to Counter Electrode) is provided at this connector. Impedance is 1000 ohms.
- (4) TRIG. IN A & B: These connectors are in parallel with the corresponding front-panel connectors.
- (5) ELECTROMETER OUT: In parallel with frontpanel ELECTROMETER MONITOR connector.
- (6) EXT. IN A & B: In parallel with front-panel EXT. SIG. INPUT connectors.
- (7) I/E OUT: Parallels I OUT connector of Model 176 or 179 except that this connector disconnects when Mode switch is set to NULL or DIRECT. When operating with M376, fullscale out at this connector is five volts as opposed to one volt at the M376 I OUTPUT connector.
- (8) ACCESS. POWER: Provides ± 24 V regulated power for accessory equipment. Up to 200 mA may be safely drawn from these sources. Pin A is ground. Pin B is +24 V, and pin C is - 24 V. The other pins are unused.

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SUMMING AMPLIFIER PERFORMANCE SPECIFICATIONS

OUTPUT VOLTAGE: At least ± 100 V. OUTPUT CURRENT: At least ± 1 A.

RISETIME (10% to 90%): At ×1 gain, closed loop and unloaded, 1 µs. If loaded at 1 A, risetime increases to 2 us.

SLEW RATE: 10 V/µs.

STABILITY: Better than 100 μ V/day (after warmup) and 20 µV/°C.

NOISE AND RIPPLE: Less than 50 µV rms.

LINE VOLTAGE SENSITIVITY: Less than 100 μV change at amplifier output for ± 10% change in line voltage.

ELECTROMETER (MODEL 178)

Each Model 173 is provided with a Model 178 Electrometer Probe. The probe must be used in Controlled Potential operation to monitor the Reference Electrode, and can be used at any time to monitor some potential of interest. To allow the electrometer circuitry to be positioned as close as possible to the monitored point, and thereby achieve minimum loading of high impedance electrodes and optimum loop stability in closed loop operation, the Electrometer Probe is located at the end of a cable that connects at the front panel of the M173. The electrometer performance specifications are:

INPUT VOLTAGE: ± 10 V max. INPUT CURRENT: 5 pA max.

INPUT CAPACITANCE: 5 pF (typical).

FREQUENCY RESPONSE: 10 MHz (1 kΩ source

impedance).

STABILITY: Better than 100 μ V/day (after warmup). TEMPERATURE COEFFICIENT: Less than 20 μV/°C.

SIZE: 17" $W \times 7$ " $H \times 15$ " D (43 cm $W \times 18$ cm $H \times 38$ cm D).

WEIGHT: 60 lbs (27 kg), including plug-in.

ACCESSORIES (OPTIONAL)

- (1) 178/41 NOISE FILTER: One source of noise in an electrochemical system is power line pickup at the cell. At high sensitivities, such pickup can cause overload and loss of potentiostatic control. A small capacitor connected between the Counter and Reference Electrodes will correct this problem. The 178/41 Noise Filter consists of a suitable capacitor (100 nF) mounted in a plastic case with a clamp that allows it to be attached directly to the M178.
- (2) 173/42 SLOW SWEEP ACCESSORY: This device is a low-cost voltage programmer used to generate voltage ramps useful for programming the potentiostat in corrosion studies. The accessory incorporates a motor-driven potentiometer with an output of 1 V, 2 V, or

5 V. Scanning times are switch-selectable in 1-2-5 sequence from one minute to 100 minutes with a ramp linearity of 0.1%. Thus scan rates are available over a range of 1 mV/minute to 5 V/minute. A front-panel switch allows the choice of ascending-ramp, descending-ramp, or ramp-arrest operation.

(3) MODEL 175 UNIVERSAL PROGRAMMER: A versatile precision programmer able to provide pulse or ramp outputs. Scan rate is adjustable from 1 mV/s to 10^4 V/s. Pulse widths are adjustable from $100~\mu s$ to 10^3 s. As many as four different inflection points can be independently selected for a single output waveform.

1.3 PLUG-IN MODULES

Although this manual is for the most part restricted to a treatment of the mainframe alone, the specifications for the modules are nevertheless included so that the operator can appreciate the increased capabilities of the M173 when it is equipped with one of the plug-in modules. In many laboratories, the number of applications for which the M173 can be used will be significantly increased through the acquisition of one or more of these modules.

1.3A MODEL 176 CURRENT-TO-VOLTAGE CONVERTER

The Model 176 is the "basic" plug-in module. It gives the M173 the capability of meeting the requirements of most potentiometric and galvanometric applications at moderate cost. A Model 173 equipped with a Model 176 can do CONTROL I operation at currents as high as 1 A. It also allows the current to be continuously monitored in CON-TROL E operation. Whether operating in the CON-TROL I mode, or monitoring the cell current in CONTROL E operation, the M176 provides a dc output voltage proportional to the full-scale current, where the full-scale current is given by the setting of the M176 RANGE switch. Full-scale current ranges from 1 μ A to 1 A can be selected. The cell current can be displayed on the panel meter at any time, with full-scale meter deflection corresponding to 1, 2, or 5 times the selected current range. There is also provision for IR COMPENSA-TION of the cell resistance between the Working Electrode and the Reference Electrode. An OVER-LOAD light indicates operation outside the normal range. Specifications follow.

- (1) CURRENT RANGES: Front-panel Current Range switch gives choice of seven full-scale ranges from 1 μ A to 1 A.
- (2) CURRENT RANGE TOLERANCE: From 1 μ A to 10 mA, tolerance is 0.1% \pm 100 ppm/°C max. From 100 mA to 1 A, tolerance is 0.2%

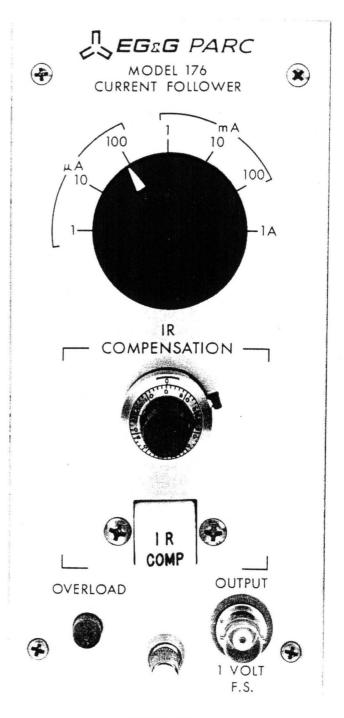


Figure I-2. MODEL 176 CURRENT-TO-VOLTAGE CONVERTER

 \pm 100 ppm/°C. Tolerance is adjustable to 0.1% on 100 mA and 1 A ranges for operation with specific M173.

- (3) OUTPUT: Voltage proportional to cell current is provided at front-panel connector. Full-scale current gives 1 V out. Unit functions properly with outputs as high as 5 V (current five times full scale) except on 1 A range where limit is 1 A.
- (4) CURRENT MONITOR: Current as a function of selected full-scale Current Range can be

displayed on the M173 panel meter by setting M173 Meter switch to CURRENT.

- (5) DIGITAL LOGIC: Also available is a Model 176D, which differs from the Model 176 only in that it provides digital Current Range data out. This BCD data is available at the M173 rear-panel Digital Interface connector if the M173 is equipped with the digital readout option.
- (6) IR COMPENSATION: A high-resolution tenturn dial allows positive feedback to be applied to the input of the Potentiostat to compensate for the resistance between the Working Electrode and the Reference Electrode. An internal switch increases the available compensation voltage by a factor of ten, sufficient to fully compensate all but extremely high resistance solutions.
- (7) CHOPPER-STABILIZED AMPLIFIER SPECIFI-CATIONS: INPUT LEAKAGE CURRENT: 10 pA max. OUTPUT VOLTAGE: 5 V max. STABILITY: Better than 10 μV/day and 1 μV/°C. NOISE AND RIPPLE: 0.05 μA max. OUTPUT CURRENT: 1 A max.

1.3B MODEL 179 COULOMETER

INTRODUCTION

The Model 179 Digital Coulometer is a plug-in module designed for use with the Model 173 Galvanostat/Potentiostat. Together they form a stateof-the-art system for making precision coulometric measurements at either a controlled potential or current. Measurements of solutions containing extremely small samples are possible. Quantities from 10 picoequivalents to 10 equivalents can be measured coulometrically with an absolute accuracy better than 0.1% of full scale and with a reproducibility better than $\pm 0.02\%$ of full scale. Although the Model 173/179 System is highly versatile, it is easy to operate, and is thus as well suited to routine measurements as it is to complex analyses. The drift of the coulometer is so low that experiments lasting hours or even days can be easily accommodated. A four-digit plus polarity and exponent digital display continuously indicates the accumulated coulombs. Automatic subtraction of any background current is provided by means of a Background Current adjustment.

DESCRIPTION

Functionally, the Model 179 can be divided into three main parts. The first is a current-to-voltage converter that monitors the cell current and provides a voltage proportional to it. The full-scale sensitivity of this converter can be set from 1 μ A to 1 A by means of a front-panel switch. The voltage out of the converter is available at the front-panel

I OUT connector. This voltage is also applied to a voltage-to-frequency converter and counter that provide the coulombic accumulation data. Last is the digital readout circuitry, which provides a fourplace display plus polarity and exponent. There is provision for automatic ranging over seven orders of magnitude. For a given range as indicated by the exponent, the four-digit display can take values from 0.000 to 9.999. The next higher count resets the display to 1.000 and advances the exponent by one digit. A built-in digital-to-analog converter produces a voltage out proprotional to the indicated coulomb accumulation. A digital output connector is also provided to facilitate automatic data recording or computer processing of the signal, if desired.

GENERAL SPECIFICATIONS

- (1) INPUT: Cell current. Front-panel switch allows selection of full-scale current ranges from 1 μA to 1 A in decade steps. Full-scale current is defined as that current which will fill the display (0.000 to 9.999) in one second (after resetting but before autoranging).
- (2) CURRENT OVERLOAD CAPABILITY: Currents as high as five times that selected by the Current Sensitivity switch can be handled without any performance degradation. This five-times overload capability applies for all but the 1 A range, where the maximum allowable current is 1 A.
- (3) DISPLAY: The display is in two portions. The upper, which consists of four digits preceded by a polarity symbol, can take values from 0.000 to 9.999 (decimal point is always fixed). Positive polarity indicates accumulation of anodic current in coulombs; negative indicates cathodic current accumulation. (In bipolar notation with current reversals, polarity indicates whether the net coulomb accumulation has been anodic or cathodic.) The lower part of the display consists of the letter E (indicates exponent to base 10) followed by a polarity sign and a digit that indicates the exponent magnitude. The display is direct reading in coulombs.
- (4) AUTORANGING: Autoranging is accomplished by advancing the exponent one digit and simultaneously down ranging the display from 9.999 to 1.000. Each autorange step represents a ten-fold decrease in sensitivity. That is, for a given input current, the rate at which the display fills decreases by a factor of ten each time the autorange action occurs.
- (5) MANUAL RESET: Positive-action toggle switch allows the display to be reset to 0.000 EX, where X is the exponent corresponding to the selected full-scale sensitivity. Example: Resetting with Current Sensitivity switch set

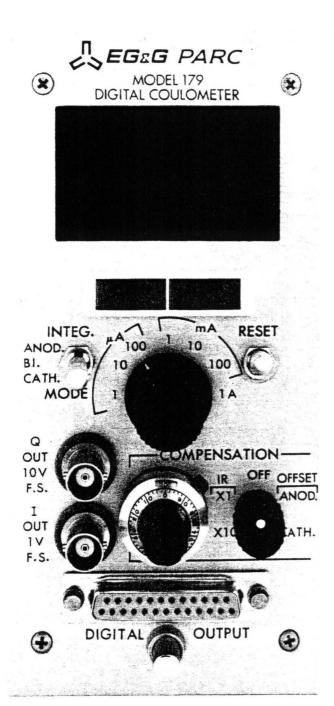


Figure I-3. MODEL 179 COULOMETER

to 100 mA will give display of 0.000 E-2. Note that a full-scale current (100 mA) will fill the display (advance it from 0.000 E-2 to 9.999 E-2; next count to autorange to 1.000 E-1) in one second.

(6) INTEGRATION MODES: Positive-action toggle switch gives choice of having integrator respond to Anodic currents only, to Cathodic currents only, or to both, in which mode the display indicates the net coulomb accumulation.

- (7) BACKGROUND COMPENSATION: Front-panel controls background currents as high as ± 10% of the selected Current Sensitivity to be nulled. These same controls can be used for positive feedback IR Compensation when desired.
- (8) INPUT CURRENT DRIFT: Less than 10 pA/day.
- (9) ACCURACY: 0.1% of full-scale display range for input currents from 10% to 500% of selected full-scale current sensitivity.
- (10) RESOLUTION: ±0.01% of full-scale display range.

(11) OUTPUTS:

I OUT: Front-panel BNC connector gives analog voltage proportional to cell current. Full-scale current gives one volt out. Polarity is positive for anodic current, negative for cathodic. Output resistance is $100~\Omega$.

Q OUT: Front-panel BNC connector gives analog voltage proportional to the three most significant digits of the four-digit display indication. Full-scale display indication gives 10 V out. Polarity is that preceding four-digit display. Exponent has no bearing on this output. Output current limit is 10 mA (1 k Ω load).

DIGITAL OUTPUT: Complete display data including polarity and exponent are provided at front-panel connector. Positive logic with BCD coding is employed. Logic 1 is $\pm 3.5 \text{ V} \pm 1 \text{ V}$. Logic 0 is 0.2 V $\pm 0.2 \text{ V}$. Table I-1 shows the connector pin assignments.

- (12) POWER: Model 179 takes its power from the Model 172 with which it is operated.
- (13) SIZE: 3" $W \times 634$ " $H \times 12$ " D (7.6 cm $W \times 17.3$ cm $H \times 30.5$ cm D).
- (14) WEIGHT: 3 lbs (1.4 kg).

CURRENT-TO-VOLTAGE CONVERTER SPECIFICATIONS

- (1) FULL-SCALE CURRENT RANGES: 7 ranges from 1 μ A full scale to 1 A full scale.
- (2) INPUT VOLTAGE DRIFT: Less than 10 μ V/day.
- (3) INPUT CURRENT DRIFT: Less than 10 pA/day.
- (4) TEMPERATURE DRIFT: Less than 0.005% of full scale per degree Celsius.
- (5) FULL-SCALE ACCURACY: 0.05%. **NOTE:** To achieve this accuracy on the 100 mA and 1 A

SIGNAL	PIN
2º of most significant digit. 2¹ of most significant digit. 2² of most significant digit. 2³ of most significant digit.	
2º of second most significant digit. 2¹ of second most significant digit. 2² of second most significant digit. 2³ of second most significant digit.	
2° of third most significant digit. 2¹ of third most significant digit. 2² of third most significant digit. 2³ of third most significant digit.	12
2º of least significant digit. 2¹ of least significant digit. 2² of least significant digit. 2³ of least significant digit.	16
Four-digit display polarity (logic 1 is $+$; logic 0 is $-$)	1
Exponent polarity (logic 1 is +; logic 0 is -)	18
2º of exponent. 2¹ of exponent. 2² of exponent. 2³ of exponent.	21
External Reset (logic 0; equivalent to activating Reset toggle switch)	24
V/F Output or Inhibit (logic 0).	25
Print Command Input	23

current ranges the unit must be factory calibrated with the Model 173 with which it is to be used.

(6) CURRENT MONITOR: Front-panel BNC connector. Full-scale current gives 1 V out.

Table I-1. DIGITAL OUTPUT CONNECTOR PIN ASSIGNMENTS

VOLTAGE-TO-FREQUENCY CONVERTER

10

- (1) FREQUENCY LINEARITY: Better than 0.1% of reading for input currents in the range of 10% of full scale to 500% of full scale. Better than 1% of reading for input currents in the range of 1% of full scale to 10% of full scale.
- (2) FREQUENCY DRIFT WITH TEMPERATURE: Less than 0.0005%/°C.
- (3) FREQUENCY ACCURACY, FULL SCALE: 0.05%.
- (4) INTEGRATION ACCURACY, COULOMBS: 0.1% of full scale for input currents in the range of 10% of full scale to 500% of full scale (±10 counts).
- (5) INTEGRATION REPRODUCIBILITY, COULOMBS: 0.02% of full scale (±2 counts).

(6) FREQUENCY: Frequency is proportional to input current. Full-scale current frequency is 10 kHz.

1.3C MODEL 376 LOGARITHMIC CURRENT CONVERTER

The Model 376 is ideally suited to use in those applications where the current varies over many orders of magnitude in the course of the experiment. Like the Model 176, the Model 376 provides an output voltage proportional to the cell current. However, it additionally provides an output voltage proportional to the *log* of the cell current. There is provision for displaying the current or the log of the current on the M173 panel meter. In displaying the current, full-scale meter deflection indicates a current of 1, 2, or 5 times the selected Current Range, the same as for the M176. In the

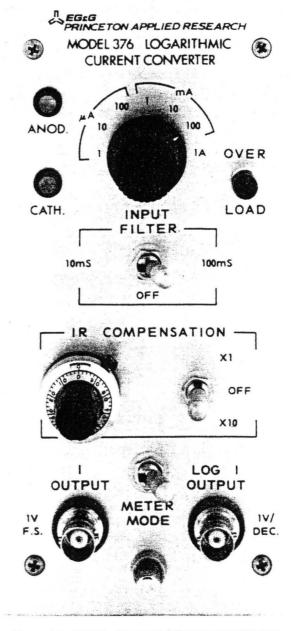


Figure I-4. MODEL 376 LOGARITHMIC CONVERTER

log Meter mode, full-scale deflection indicates a current 1, 2, or 5 decades below the selected current range. For example, a full-scale current will give a 0 meter indication. A current of 0.1 full scale will give a - 1 V meter indication. A current of 0.01 full scale will give a - 2 V meter indication, and so forth, to a maximum of -5 V (Meter Sensitivity to 5) for a current of 10⁻⁵ full scale. The transfer function to the LOG I OUT connector is 1 V per decade. A current equal to the selected range will give 0 V out. A current equal to the selected range will give 0 V out. A current of 0.1 full scale will give +1 V out, and so forth down to a current of 10-5 full scale which will give +5 V out. Other features include an input filter controlled by a front-panel switch, and full IR COMPENSATION capability like that provided with the M176.

When operated in conjunction with a M376, the M173 can easily perform such difficult studies as corrosion measurements, battery or fuel-cell testing, and electrode materials evaluators. All factors considered, the M376 offers the most overall system versatility obtainable with a single plug-in module.

SPECIFICATIONS

- (1) CURRENT RANGES: Front-panel Current Range switch gives choice of seven full-scale ranges from 1 μA to 1 A.
- (2) ACCURACY: 0.2% over five decades.
- (3) METER MODES: Positive-action toggle switch gives choice of two meter modes. In I MODE, meter gives linear indication of cell current relative to selected CURRENT RANGE. In LOG I MODE, meter indicates log of cell current relative to selected CURRENT RANGE.

(4) OUTPUTS:

I OUTPUT: Voltage output proportional to current. Full-scale current gives 1 V out. Maximum output is 5 V (except on 1 A range, where 1 A maximum current capability limits output to 1 V).

LOG I OUTPUT: Voltage proportional to the log of the cell current relative to the selected range. Transfer function is +1 V/decade. Maximum is +5 V.

DIGITAL RANGE: Current Range data in BCD format is provided at M173 rear-panel Digital Interface connector if M173 is equipped with Digital Panel Meter.

(5) INPUT FILTER: Positive-action front-panel switch gives choice of input filtering time constants of 10 ms or 100 ms. An OFF position is also provided. (6) IR COMPENSATION: A high-resolution tenturn dial allows positive feedback to be applied to the input of the Potentiostat to compensate for the resistance between the Working Electrode and the Reference Electrode. An associated switch allows the available compensation to be increased by a factor of ten, sufficient to fully compensate all but extremely high resistance solutions.

1.4 MAINFRAME DESCRIPTION

As shown in Figure I-5, the basic Model 173 mainframe consists of two potential sources, some control circuits to switch the potential sources and determine their output level, and a high-power high-speed Summing Amplifier. Figure I-1 shows how the Model 173 mainframe would be used in a CONTROL E application (CONTROL I operation is impossible with just the mainframe). A potential, either internally derived or derived from an external source connected to one of the two EXT SIG IN connectors, is applied to the input of the Summing Amplifier. The Summing Amplifier output is applied to the Counter Electrode of a cell. The Working Electrode is grounded (the necessary connections are made by the "dummy" plug which must be installed to operate the mainframe by itself). The Model 178 Electrometer Probe monitors the potential at the Reference Electrode and feeds it back to the input of the Summing Amplifier. The panel meter indicates the Reference Electrode Potential (meter polarity is actually such as to directly indicate the Working Electrode potential with respect to the Reference Electrode). The system comes to equilibrium when the feedback potential (the potential at the Reference Electrode) is equal and opposite to the selected potential, and the Summing Amplifier output drives the Counter Electrode to whatever potential is required (within the ± 100 V compliance capabilities of the amplifier) to make this happen.

For example, if the selected potential were +1 V, the Counter Electrode would be driven as far negative as is necessary to make the Reference

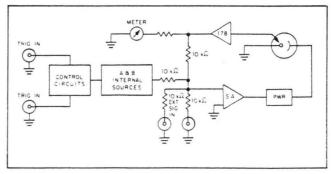


Figure i-5. MODEL 173 MAINFRAME IN CONTROL E OPERATION (no plug-in, "dummy" connector installed)

Electrode – 1 V with respect to the Working Electrode (ground). Otherwise expressed, the Counter Electrode is driven to where the Working Electrode is at the applied potential (+1 V) with respect to the Reference Electrode, and the meter will indicate +1 V. Thus the Working Electrode is at the programmed potential with respect to the Reference Electrode, even though, in absolute terms, the Working Electrode is always at ground, and a voltage of the opposite polarity is applied to the Counter Electrode.

1.5 CONTROL E AND CONTROL I OPERATION WITH A MODEL 176

Figure I-6 illustrates how the Model 173 functions in Control E and Control I operation with a Model 176 Plug-In Module. Referring to Figure I-6a, note that in Control E operation the selected potential is applied through a 240 k Ω resistor to the summing junction of the potentiostat. At the same time, the potential between the Working Electrode and the Reference Electrode is detected by the Model 178 Electrometer Probe and also fed back to the summing junction. Loop stability criteria dictate that the potential at the summing junction be 0 V, and the potentiostat output will automatically drive the Counter Electrode to whatever potential is required to establish and maintain equilibrium. In other words, the potential across the Working and Reference Electrodes will automatically be maintained at the selected potential (assuming the necessary Counter Electrode potential is somewhere in the range of $\pm 100 \text{ V}$). Usually the operator will be interested in the Cell current (determined by electrolyte characteristics and Counter Electrode potential). As shown, the

Model 176 operates as a current-to-voltage converter to provide an output voltage proportional to the cell current, allowing it to be measured on the panel meter or by other means.

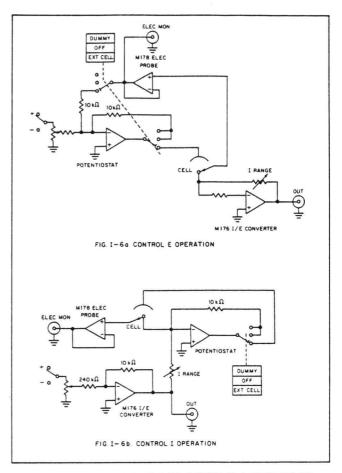


Figure I-6. CONTROL E AND CONTROL I OPERATION WITH A MODEL 176 PLUG-IN MODULE

SECTION II CONTROLS

2.1 INTRODUCTION

This section is included to help familiarize the operator with the placement of the controls and to provide a brief description of their functions.

2.2 FRONT-PANEL CONTROLS AND INPUTS (see photo on page I-1)

2.2A APPLIED POTENTIAL/CURRENT

This section contains the controls which determine the internally-selected potential or current to be applied to the cell, as well as the external control signal inputs and the inputs and switching for selection of the applied control signal.

(1) Potential/Current Controls

The toggle-switch, rotary-switch, vernier combinations located in the A and B bracketed areas are 4-digit direct-reading controls for setting the potential or current to be applied. The polarity of the signal is controlled by the toggle switch, with the central position on each switch corresponding to zero applied signal. The rotary switch and vernier potentiometer, when read directly across a line, indicate the voltage to be applied (Figure II-1). Thus, if the toggle switch was in the minus position, the rotary switch in the 1 position. and the vernier at 239, the applied potential selected would be - 1.239 volts. In CONTROL I operation with a Model 176, 179, or 376 these controls set the current supplied relative to that selected by the Current Range switch.

(2) Pushbuttons and Associated Toggle Switch

Four lighted pushbutton switches and an associated miniature toggle switch determine which of the internal sources drives the Summing Amplifier, and control the switching back and forth between these two sources. The left-most button, labeled ZERO I, is used only in CONTROL I operation. In the ZERO I state, the cell current is reduced to less than .01% of the selected full scale value.

When the A button is depressed, the potential or current selected by the A set of controls is applied. Similarly, when the B button is depressed, the value selected by the B controls is applied. It should be noted that the ZERO I, A, and B pushbuttons are momentary-contact switches and should not be held in.

When the external trigger pushbutton is depressed, external signals fed into the two

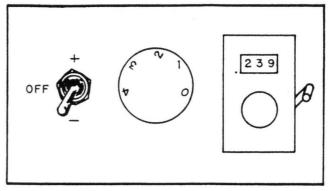


Figure II-1. CONTROL SETTINGS FOR "A"
POTENTIAL OF -1.239 V

TRIG. INPUT BNC connectors on the front panel cause the unit to switch from source A to source B and back in a variety of ways, as described below. The instantaneous state of the control signal may be ascertained by noting which of the pushbuttons is glowing. Each glows when the corresponding source controls the Summing Amplifier.

Three modes of external triggering are available. When the control switch is in the REPET. position, an incoming trigger signal causes the unit to switch from one source to the other. The polarity of the control signal which causes the transfer to occur is determined by which of the two TRIG. INPUT BNC's is used. The left BNC responds only to a positive-going edge, and the right only to a negative-going edge.

In the SINGLE position, only one transfer takes place. Afterwards, additional trigger signals into that input have no effect (unless severe overdrive conditions occur). A-to-B transfers can be effected at the left input only. Similarly, B-to-A transfers can be effected at the right input only. The pushbuttons are always active.

In the TRACK position, the two inputs are tied together so that transfer takes place on either a positive-going or a negative-going edge applied to either input.

As an illustrative example of the operation of the triggers, envision a system where a square wave is applied to one trigger input or the other. In the REPET. mode, the output signal of the potentiostat would be a square wave of exactly 1/2 the frequency of the incoming signal, since it would only respond to positive-going edges or negative-going edges, according to which Trigger Input the

signal was applied to. In the TRACK mode, however, a square wave at the same frequency would be obtained, since both inputs would be sensitive to both positive-going and negative-going signals.

The two EXT. SIG. INPUT connectors and the associated toggle switches accept inputs which are added to the output of whatever source is selected by the internal programming circuitry. Either or both of these external signal inputs may be used at any time, and the control signal applied to each of these BNC's will be added to whatever signals are determined by the internal programming.

2.2B OPERATING MODE

(1) Operating Mode Switch

The four position OPERATING MODE rotary switch selects the specific technique to be performed by the instrument. In the CONTR. E mode, the instrument acts as a potentiostat, causing the potential selected by the applied Potential/Current controls to be maintained between the working and reference electrodes by varying the voltage applied to the counter electrode. CONTROL E operation is possible with any of the plug-in modules, or even with the mainframe alone.

In the two MEASURE ONLY modes, NULL and DIRECT, the instrument monitors the voltage at the Reference Electrode with respect to ground, and displays the potential of the Working Electrode with respect to the Reference Electrode. The circuit to the Counter Electrode is open in both of these modes so that there is no cell current in either case.

In the DIRECT mode, the potential of the Working Electrode with respect to the Reference Electrode is read directly from the meter, with full-scale meter indication being 1, 2, or 5 V, according to the setting of the Sensitivity knob of the Meter switch. NOTE: Selector switch must be set to EXT. CELL and Meter Function switch to POTENTIAL.

In the NULL mode, the Applied Potential/Current controls are adjusted for null indication on the meter, after which the potential of the Working Electrode with respect to the Reference Electrode is read from the setting of the Applied Potential/Current controls. High resolution readings are thus possible. As with the DIRECT mode, the Selector switch must be set to EXT. CELL and the Meter Function switch to POTENTIAL. Note that a NULL measurement is not a null-balance bridge measurement. The extremely high input resistance (10¹¹ ohms) of the electrometer pre-

cludes the necessity for bridge measurement techniques.

In the CONTR. I mode, the instrument acts as a galvanostat, maintaining a constant current between the Working and Counter Electrodes. The electrometer is not in the control loop, but can still be used to measure the potential at a selected point. CONTROL I operation is only possible if the M173 is operated with a plug-in module.

(2) Selector Lever Switch

The SELECTOR lever switch allows the instrument to be connected to the external cell, or to an internal dummy cell, a precision $10~\mathrm{k}\Omega$ resistor, which allows testing of instrument function without regard for problems which may be introduced by the external cell. An OFF position is also provided in which the potentiostat output is disconnected. It should be noted that, in CONTR. I operation, the current level indication (panel or other) continues even if the Selector is set to DUMMY CELL or OFF. This "current" is internal to the M173 and does not flow through the cell. Cell current is obtained *only* when the selector is set to EXT. CELL.

(3) Connectors

Two connectors are provided in this section of the front panel. Both are multi-pin quick disconnect sockets, and the two are different to prevent one from connecting the wrong cable to either connector. The External Cell connector interconnects the M173 and the cell. This cable must be connected for proper operation with a plug-in module (to reduce IR drop errors, the input summing junction of the module is brought out, via this cable, to the cell). The Electrometer Probe connector accepts the output of the Model 178 Electrometer Probe and also provides the \pm 24 V and ground connections for its power.

2.2C READOUT SECTION

(1) Overload Light

The Overload light comes on when any one of the instrument amplifiers is driven to its limit, or when a malfunction in one of the power supplies causes an amplifier to remain at a limiting potential. Occasional flickers of the Overload light, especially when the applied potential is suddenly stepped, simply mean that the full current is being drawn from the instrument momentarily, and should not be a matter for concern. If the light remains on, however, something is probably wrong and the instrument should be checked.

(2) Meter and Switches

The analog meter normally supplied with the M173 is a precision device fitted with three scales. A dual concentric switch directly below this meter determines the sensitivity and whether the quantity displayed is potential or current.

When the outer knob is set to POTENTIAL (SELECTOR to EXT. CELL), the potential of the Working Electrode with respect to the Reference Electrode is displayed, with full scale meter indication being 1 V, 2V, or 5 V, according to the position of the inner knob. This is true whether the instrument is operating in the CONTR. E or CONTR. I mode. In CONTROL I operation, if the Electrometer Probe is not connected (and it need not be), the display indication will have no significance. NOTE: When Meter switch is set to POTENTIAL. Cell Selector switch must be set to EXT. CELL to obtain a meaningful meter indication. This is true for all operating modes.

When the outer knob is set to CURRENT (M173 must be equipped with a plug-in module), the meter indicates the current level relative to the current selected with the Current Range switch (each module has a Current Range switch). With a M176 or M179, fullscale meter deflection will correspond to 1, 2, or 5 times the Current Range switch setting, according to the position of the inner knob of the Meter switch. The M376 is a special case as explained in the M376 specifications (Subsection 1.3C).

In the case of a unit equipped with the digital readout option, the "1" and "2" full-scale ranges are identical, with a maximum possible display indication of 1.999. Full-scale indication on a "5" range is 5.00.

(3) Electrometer Monitor BNC Connector

The voltage monitored by the Model 178 Electrometer Probe is provided at the front-panel ELECTROMETER MONITOR connector and at the rear-panel ELEC. OUT connector. The two connectors are in parallel and the source resistance at each is 1 k Ω . The potential at these connectors can be monitored with an oscilloscope or some other external monitoring instrument. Note that whenever the Meter switch is set to POTENTIAL, the meter will indicate the magnitude of the voltage at the ELECTROMETER MONITOR connector, but the opposite polarity (exception: NULL mode). The potential at the ELECTROMETER MONITOR connector will be that of the Reference Electrode with respect to ground. The meter indicates the potential of the Working Electrode with respect to the Reference Electrode (the Working Electrode is always at ground potential). If the Model 178 Electrometer Probe is not being used to monitor some potential point, such as could be the case in CONTR. I operation, the potential at the ELECTROMETER MONITOR connector will have no significance.

2.3 REAR-PANEL CONNECTORS

All of the BNC connectors present on the front panel are also brought out to the rear panel. In addition, other connectors, as described below, are provided.

TRIG-OUT: These two connectors, labeled A and B, provide outputs suitable for triggering external monitoring instruments such as oscilloscopes. A positive 1 volt level is available at the A connector whenever the A button is depressed or the A potential is selected by an external trigger. The 1 volt level is available at the B connector whenever the B button is depressed or the B potential is triggered. When the corresponding source is not active, ground is available.

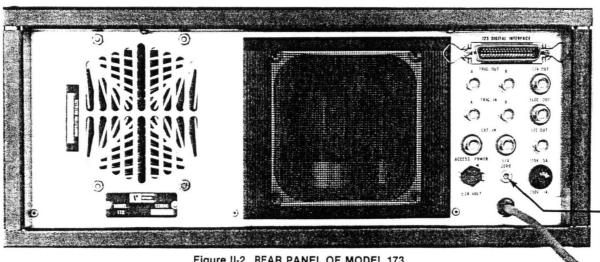


Figure II-2. REAR PANEL OF MODEL 173

11-3

S/A OUT: This is the output of the control amplifier and shows the voltage applied to the Counter Electrode. It is the actual control amplifier output.

TRIG-IN A & B: These connectors are in parallel with the corresponding front-panel connectors.

ELECT-OUT: In parallel with front-panel ELECTROMETER MONITOR connector.

EXT. IN: In parallel with front-panel EXT. SIGNAL INPUT connectors.

I/OUT: Low impedance 1 V f.s. output voltage corresponding to front-panel I OUTPUT signal in

M176 and M179. In M376, full-scale output is five volts at this connector. This output is disconnected in DIRECT and NULL operating modes.

ACCESS. POWER: Provides ± 24 V regulated power for accessory equipment. Up to about 200 mA may be safely drawn from these sources. Pin A is ground. Pin B is + 24 V, and pin C is - 24 V. The other pins are unused.

S/A ZERO: This screw adjustment is used to zero the control amplifier as outlined in the Calibration Procedure.

DIGITAL INTERFACE: See Table IV-2 on page IV-5.

SECTION III INSTALLATION PROCEDURE AND INITIAL PERFORMANCE CHECKS

3.1 INSTALLATION

No special installation requirements need be met, except that the rear panel should be reasonably accessible. The monitoring connectors are located at the rear panel. Access to it may be necessary when conducting experiments where external inputs are being supplied to the instrument, or where outputs from the instrument are being used for external triggering or display. Access to the monitor panel is also necessary for troubleshooting, and occasional access to the power takeoff connectors may be required. In addition, the rear panel contains air intake and exhaust ports which should not be blocked.

3.2 POWER REQUIREMENTS

The Model 173 will accept either 105-125 or 210-250 V ac. 50-60 Hz, and requires about 175 voltamperes of power. The operating voltage range is selected by an internal switch located just to the front of the power transformer. Instructions for setting this switch are provided in the Safety Instructions at the beginning of the book. Units are normally sent with the switch set to "115", the proper setting for operation from voltages in the 105-125 V range. In the case of a unit shipped with the switch set to "230", the setting for operation from voltages in the 210-250 V range, a red tag labeled "220 V" is normally attached to the line cord. FOR OPERATOR SAFETY, THE SETTING OF THE LINE VOLTAGE SELECTOR SWITCH SHOULD ONLY BE CHECKED OR CHANGED WITH THE UNIT DISCONNECTED FROM ALL SOURCE OF POWER. AS DESCRIBED IN THE SAFETY INSTRUCTIONS, THE CHECK (CHANGE) PROCEDURE SHOULD ONLY BE PERFORMED BY QUALIFIED SERVICE PERSONNEL.

3.3 FUSING

The ac power fuse is located at the rear panel. For operation from a line voltage of nominally 115 V, use a slow-blow 5 A fuse with a voltage rating of 125 V or higher. For operation from a line voltage of nominally 230 V, use a slow-blow 3 A fuse with a voltage rating of 250 V or higher.

In addition, there are six internal fast-blow fuses that protect the dc supplies. The current rating of these fuses is indicated in Figure V-1 on page V-1. In each case, use a fast-blow fuse with a voltage rating equal to, or higher than, the voltage indicated in Figure V-1.

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

WARNING!

TO AVOID THE POSSIBILITY OF A SAFETY HAZARD FROM ELECTRICAL SHOCK, WHICH COULD RESULT IN PERSONAL INJURY OR DEATH, DISCONNECT THE POWER CORD BEFORE REMOVING OR INSTALLING A FUSE. FUSES SHOULD ONLY BE CHANGED BY QUALIFIED SERVICE PERSONNEL.

3.4 CABLING

The M173 is furnished with two special interconnecting cables to interface the instrument with standard electrochemical cells. The two cables are:

(1) A four-foot, 6-conductor multi-shield cable fitted with one male 6-pin plug and terminated in 3 color-coded crocodile clips. This cable connects the Working and Counter Electrodes to the instrument. The color code is:

> Red—Counter Electrode Green—Working Electrode Black—Ground

NOTE: When a plug-in module is used, the summing junction of the module Input Amplifier is brought out into the cable to minimize IR drop errors. It is therefore essential to have this cable plugged in before using the instrument. Otherwise the plug-in module will not function properly.

WARNING! When operating in the CONTROL E or CONTROL I mode, voltages as high as 100 V with a current capability of 1 A may be present at the external cell cable alligator clips when the Selector switch is in the EXT. CELL position. To minimize the possibility of dangerous, possibly lethal electrical shock, operators are warned not to touch these exposed clips, either to connect to a cell, or to disconnect from one, unless the Selector switch is first placed in the OFF position.

(2) In addition, the Model 178 Electrometer Probe must be connected in CONTR. E operation. The Electrometer Probe connector on the front panel provides ± 24 volts and ground to the probe, and also carries the probe output signal back to the Model 173. For proper operation in the CONTR. E mode with the Selector set to EXT. CELL, the probe must be connected to the Reference Electrode at the cell, although an external load resistor may be substituted for the cell to perform certain checks.

3.5 INITIAL PERFORMANCE CHECKS

3.5A INTRODUCTION

The following procedure is provided to facilitate initial-performance checking of the Model 173 Potentiostat/Galvanostat. In general, this procedure should be carried out after inspecting the instrument for shipping damage and reporting any such damage to both the carrier and to EG&G PRINCETON APPLIED RESEARCH, but before attempting to use the instrument experimentally. No special equipment is needed. NOTE: THESE CHECKS APPLY TO A MAINFRAME OPERATED ALONE. FOR CHECKS WITH A PLUG-IN MODULE, SEE THE CORRESPONDING PLUG-IN MODULE INSTRUCTION MANUAL. Should any difficulty be encountered in carrying out these checks, contact the factory or the factory-authorized representative in your area.

3.5B PREPARATION

- (1) After examining the instrument for signs of visible damage, and before plugging in the line cord, loosen the two screws on the underside of the rear overhang of the top cover and remove them. Then slide this cover backward until the interior of the instrument is clearly visible. Check to insure that all three circuit boards are firmly seated in their sockets and that the cable that extends from the heat-sink assembly to the chassis connector just in front of the assembly is plugged in. Then slide the cover back in and replace the screws.
- (2) Plug in the "dummy" plug-in mating connector and install the blank panel. **NOTE:** These items are supplied only when a Model 173 is purchased without a plug-in module. The plug-in connector and blank panel are supplied uninstalled in a separate package. The connector is installed by pushing it into the plug-in module receptacle (it can only go in one way) as far as possible. The blank panel should then be fitted in place and tightened by means of the locking screw.

3.5C PROCEDURE

(1) With the power off, set up the controls and connections as follows.

Cell Selector switch: OFF Operating Mode: CONTR. E

"A" & "B" Potentials: OFF and 0.000 V

Ext. Sig. Inputs: OFF

Meter switch: POTENTIAL and "1" External Cell Cable: plugged in Electrometer Probe: plugged in

- (2) Connect an external 1 kilohm load resistor as shown in Figure III-1. Take care that the black clip (unused) doesn't short against another circuit element.
- (3) Turn the power on, and, if the Overload light does not come on, proceed to the next step. If the Overload light does come on, the instrument has sustained some shipping damage and either the factory or your local representative should be contacted.
- (4) The "A" pushbutton should light when the power is turned on and the meter should read zero.
- (5) Switch the Cell Selector to EXT. CELL and again note that the meter reads zero.
- (6) Set the "A" potential to +1.000 V and note that the meter deflects full scale.
- (7) Depress the "B" pushbutton and note that the meter returns to zero (the "B" light should come on).

This completes the Initial Checks. If the indicated results were obtained, one may be reasonably sure that the instrument has arrived in good working order. Bear in mind that in the case of a unit operating without a plug-in module, CONTROL I operation is impossible. Also, there is no way of measuring the cell current in CONTROL E operation.

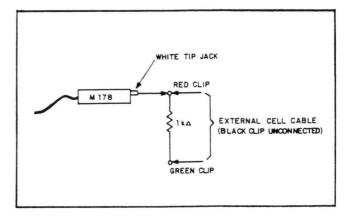


Figure III-1. EXTERNAL RESISTOR CONNECTIONS

SECTION IV OPERATING INSTRUCTIONS

4.1 INTRODUCTION

This section of the manual discusses the details of operating the mainframe by itself. Operation with any one of the plug-in modules is treated in the corresponding plug-in module manual. For a treatment of electrochemistry *per se*, the reader is referred to the bibliography at the end of this section.

Two key points must be considered in setting up the Model 173 and its associated instruments for an electrochemical experiment. First of all, the Working Electrode is maintained at either virtual or hard ground (zero volts) by the instrument. That is to say, although reference is frequently made to a potential applied to the Working Electrode, in actual fact the Working Electrode is maintained at zero volts, and an opposing potential is applied to the Counter Electrode and detected by the Reference Electrode. For example, if one desires to apply -1 V to the Working Electrode, and in fact sets the controls to do so, a measurement would show zero volts at the Working Electrode and + 1 V at the Reference Electrode (Reference Electrode potential is available at ELECTROMETER MONITOR connector in CONTROL E operation). This should be remembered to avoid confusion.

Second, one must understand the distinction between the EXT. SIG. INPUT connectors and the TRIG. INPUT connectors. Signals applied to the EXT. SIG INPUT connectors add directly to the applied potential selected with the APPLIED POTENTIAL/CURRENT controls. If the APPLIED POTENTIAL/CURRENT toggle is set to OFF, then the externally derived signals alone determine the applied potential. The inputs are non-inverting, i.e., plus one volt applied to either of the EXT. SIG. INPUTS (assuming APPLIED POTENTIAL/CURRENT is OFF) will put the Working Electrode at + 1 V with respect to the Reference Electrode.

Signals applied to the TRIG. INPUT connectors cause the instrument to internally switch back and forth between the A and B control sources. The basic requirements on these trigger signals are that they have an amplitude of at least 3 V and that their rise time be faster than 100 μs (see Subsection 4.3D).

4.2 CELL CONNECTIONS

11

Connections to the electrochemical cell are made through the external cell cable, which is fitted with a 6-pin male connector at one end and terminated in three colored crocodile clips at the other end, and through the Model 178 Electrometer Probe. Connections are as follows.

(1) External Cell Cable

Clip Color	Electrode Conn.
Red	Counter Electrode
Green	Working Electrode
Black	Ground

(2) Model 178 Electrometer Probe

Connect to the Reference Electrode, using as short a piece of wire as possible.

The active amplifier in the Model 178 Electrometer Probe is the black probe itself. In order to minimize the cable capacitance that the Reference Electrode must drive, the wire connecting the white input jack on the green face of the electrometer probe should be kept as short as possible. Ideally, the probe should be clamped next to the electrochemical cell and an extremely short length of wire should extend from the white input connector to the Reference Electrode.

The red crocodile clip is connected to the Counter Electrode of the electrochemical cell. Since large currents may flow in the Counter Electrode, it is particularly important that the Counter Electrode contact be a good one.

The green crocodile clip is connected to the Working Electrode. No matter what type of Working Electrode is used, the wire between the Working Electrode and the green clip should be kept as short as possible to minimize error from voltage drop in the wire. Like the Counter Electrode, the Working Electrode can carry heavy currents and any connections to it should be "solid".

The black clip (ground) is not connected to the cell. Generally, it is not used, although it may be effectively used to make contact to a shield surrounding the cell. When the black clip is not used, care must be taken to prevent it from shorting against any of the other circuit elements.

4.3 CONTR. E (Potentiostatic Operation)

4.3A INTRODUCTION

As mentioned previously, when the mainframe is operated by itself, it is limited to CONTROL E operation. Moreover, there is no way of monitoring the cell current when the instrument is operated in this manner. The following paragraphs treat this limited mode of operation. Detailed discussions of operation with the various plug-in modules are provided in the separate module instruction manuals.

To operate the mainframe by itself, it is necessary that a "dummy" plug be installed in the connector which otherwise mates with the plug-in module, and that the blank panel be mounted over the module opening. These items are ordinarily supplied only if the Model 173 has been ordered without any plug-in modules. They are shipped uninstalled. The dummy plug is installed by pushing it into the plug-in module receptacle (it can only go in one way) as far as possible. The blank panel should then be fitted in place and tightened by means of the locking screw.

4.3B INITIAL STEPS

Because of the high compliance capabilities of the Model 173 (\pm 100 V), for safety's sake, it is important that the SELECTOR switch be set to OFF before any further steps are taken. In this way, the possibility of accidental shock is greatly reduced. ONLY WHEN ALL PREPARATIONS ARE COMPLETED, INCLUDING CONNECTING ALL OF THE CABLES, SHOULD THE SELECTOR BE PLACED IN THE EXT. CELL POSITION. Also, since the basic premise underlying this discussion is that the mainframe is to be operated by itself, the Model switch should be set to CONTROL E.

Assuming the cell has been prepared and the Model switch and Selector set as indicated, the operator can make the cable connections to the cell. The red clip connects to the Counter Electrode and the green one to the Working Electrode. The black clip is ordinarily not used, although care should be taken to see that it doesn't short against any of the other circuit elements. The Model 178 Electrometer Probe should be connected by the shortest possible lead to the Reference Electrode. If a Model 178/41 Noise Filter is used, connect it between the Reference and Counter Electrodes, using the shortest possible leads. NOTE: In most instances, use of the filter will not be necessary.

4.3C POTENTIAL SELECTION

The two internal potential sources in the APPLIED POTENTIAL/CURRENT section of the front panel allow the selection of two different potentials. Which of the two is applied depends on which of the two pushbuttons, "A" or "B", is illuminated. The selected potential corresponding to the illuminated pushbutton will be applied to the cell when the SELECTOR switch is placed in the EXT. CELL position. If two sequential potentials are desired, each may be set on a different set of controls. They may then be switched back and forth, either by depressing the pushbuttons or by applying external triggers.

When selecting a potential, the toggle switch determines the polarity of the applied potential. The voltage, in 1 V increments, is selected by the large round dial, with the number that is aligned

with the three numbers of the vernier corresponding to the voltage to be applied. The vernier then provides a three-digit continuously variable selection of the voltage over a 1 V range. Thus, if the toggle switch is up, the round dial is at 1 V, and the vernier reads 345, then +1.345 V will be applied. An OFF position is provided for both toggle switches to facilitate setting the internal sources to zero, if desired. (Alternatively, the controls could be set to 0.000 V.) If neither pushbutton is illuminated, the instrument is malfunctioning or the light is inoperative. Again, it must be emphasized that the A & B potentials are alternative, not additive. Potentials to be added must be applied to the EXT. SIG. INPUTS (see Subsection 4.3G).

As a final note, the ZERO I pushbutton is not functional in CONTROL E operation.

4.3D EXTERNAL TRIGGERING

In addition to switching from the A potential to the B potential by the use of the front-panel pushbuttons, the instrument will switch from one potential to the other on receipt of an external triggering signal when the EXT. TRIG. pushbutton is depressed. The exact way in which the triggering signal affects the applied potential depends both on which of the two inputs is used and on the position of the associated toggle switch. These effects are outlined in the table below. The minimum triggering voltage required is ± 3 V, and the rise time must be faster than 100 microseconds. The maximum rate at which the system may be triggered is 200 kHz, from a 5 volt square wave.

TRIGGER MODE

CONTROL

Single Switches from Channel A to Channel B when positive edge is applied to left connector.

Switches from Channel B to Channel A when negative edge is applied to right connector.

Repet. Channel switches on each positive edge applied to left connector and/or on each negative edge applied to right connector.

Track Channel switches on both positive and negative edges applied to either connector.

Table IV-1. EXTERNAL TRIGGERING REQUIREMENTS

Note that in the SINGLE and REPET. positions, the left side connector responds only to positive-going signals and the right side connector only to negative-goiong signals. This permits the use of two different triggering signals, from two different sources, one connected to the right connector and the second to the left connector.

4.3E TRIGGERING BY EXTERNAL CONTACT CLOSURE

It is also possible to trigger the Model 173 by means of an external contact closure to ground. One would normally operate with a single-pole double-pole device connected so that the rotor is returned directly to ground. One stator terminal should be connected to one of the two TRIGGER INPUT BNC connectors, and the other stator terminal should be connected to the other of the two TRIGGER INPUT connectors. As the switch (or relay) is actuated back and forth between the two positions, ground will be applied first to one TRIG-GER INPUT connector and then to the other. Each actuation will trigger the Model 173 the same as if one were using the Model 173 Trigger pushbuttons or an external signal applied to the TRIGGER INPUT connectors. The only restriction is that the Model 173 MUST be operated in the SINGLE Trigger mode. The TRACK mode and REPETITIVE mode won't work.

4.3F TRIGGERING FROM THE MODEL 175

In some applications it may be desirable to trigger the Model 173 from the Model 175 FRAME SYNC. or CYCLE SYNC. output. This can be done, but only if these outputs are converted from ac coupling to dc coupling. Then either output can be used as a trigger without need for any special operating considerations. However, take care not to short the FRAME SYNC. or CYCLE SYNC. output to ground or to a low-impedance potential. Such a short could damage the Model 175 output circuits.

To convert the ac coupled Model 175 outputs to dc coupled outputs, proceed as follows.

- (1) Open up the Model 175 and remove the center circuit board. Then locate the quickdisconnect terminals J30 and J42 on the Mother board, and transfer the wire on J30 to J42.
- (2) Similarly, locate quick-disconnect terminal J29 and transfer the wire on J29 to J41.

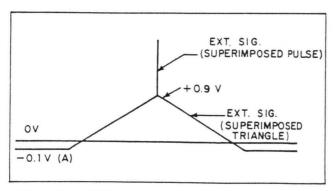


Figure IV-1. COMPOSITE WAVEFORM FROM APPLYING DIFFERENT SIGNAL TO EACH EXT. SIG. INPUT

This completes the procedure. Return the middle board to its proper operating position and close up the instrument. The signals from the Trigger outputs will now be pulses on a level and will not be differentiated as they were before.

4.3G EXTERNAL INPUTS

In addition to the fixed potential provided by the A and B potential controls on the front panel, external fixed or varying potentials may be applied to the EXT. SIG. INPUT connectors. These are voltage-control points, and have an input impedance of 10 k Ω each. The voltage applied to these connectors is added to that selected by either the A or B front-panel potential controls. If signals are connected to both EXT. SIG. INPUT BNC's, these two signals will both be added simultaneously to the potential selected on either the A or B potential controls.

Thus, for example, one could set a potential of -0.1 V on the A controls, add a triangle voltage from 0 to 1 V to the first external input, so that the result would be a 1 V amplitude triangle beginning at -0.1 V and going to +0.9 V, and superimpose on this a high speed short duration pulse at the apex of the triangle through the second input. The resultant waveform will be as shown in Figure IV-1. It is essential to remember that the inputs add so that signals applied to the External Input connectors need not be offset from ground. The applied waveform is added to the initial potential selected by the front-panel potential control. Note also that there is a toggle switch associated with each external input so that the external signal source need not be disconnected when its use is no longer required.

4.3H RUNNING THE EXPERIMENT

After insuring that the proper potentials have been selected at the front panel and that any desired additional external inputs are properly connected and switched on, the Selector switch may be set to the EXT. CELL position to begin the actual experiment. As soon as this is done, the Working Electrode will be driven to the programmed potential with respect to the Reference Electrode. (As discussed earlier, the Working Electrode is at ground potential. The desired relationship is established by driving the Counter Electrode with an opposite polarity voltage to offset the Reference Electrode by the required amount.) If the EXT. SIGNAL INPUTS are used to apply a complex control program, that program will be applied in the same manner.

The actual voltage applied to the Counter Electrode can be monitored by measuring the voltage at the rear-panel S/A OUT connector. This point is at the Control Electrode voltage whenever the SELECTOR is set to EXT. CELL. The voltage observed should be of opposite polarity to that programmed, and its magnitude will be a function

of the cell characteristics. The control voltage or program can be checked at this connector directly by setting the Cell Selector to OFF. With the switch in this position, the connection to the Counter Electrode is broken and the internal circuitry is arranged such as to allow the control voltage to be measured directly (though of opposite polarity).

The front-panel meter can be used to monitor the voltage at the Working Electrode with respect to the Reference Electrode. As long as the associated meter switch is set to POTENTIAL, the voltage at the Working Electrode with respect to the Reference Electrode is displayed on the meter. Full scale is 1, 2, or 5 V, according to the setting of the Sensitivity knob of the Meter switch. This Reference Electrode voltage with respect to ground is available at the ELECTROMETER MONITOR connector. As long as the potential is being properly controlled, the voltage at the ELECTROMETER MONITOR connector will be a faithful 1:1 reproduction of the applied control program, though of opposite polarity. The voltage at the Electrometer Monitor connector is the voltage monitored by the Model 178 Electrometer Probe. In the OFF and DUMMY CELL positions, the meter is disconnected from the electrometer output and no meter indication is obtained. The ELECTROMETER MONITOR connector, however, still connects to the Model 178 output, and still indicates the Reference Electrode potential. The stability of the voltage at this connector gives some indication of the quality of the electrode.

As a final note, a word about cell current may be in order. A Model 173 mainframe operated without a plug-in module cannot be operated in the CONTROL I mode. Moreover, there is no way even of monitoring the cell current. The cell current will vary with the applied potential according to the cell characteristics, up to a maximum of 1 A. If the cell characteristics are such as to require more current than can be supplied to establish the desired potential relationship between the Working and Reference Electrodes, the OVERLOAD light will glow.

4.31 CURRENT MEASUREMENTS

As mentioned previously, there is no way to measure the cell current (or to control the cell current) when the Model 173 mainframe is operated by itself. The unit must be fitted with one of the plugin modules to achieve this function. Once a plugin module is installed, the cell current relative to the Current Range selected at the plug-in module can be read directly from the panel meter by operating with the Meter switch in the CURRENT position.

4.3J POTENTIAL MEASUREMENTS

Measuring potential with the Model 173 involves simply selecting the method of measurement with

the Operating Mode switch, and then taking a reading. Two modes of measurement, DIRECT and NULL, are possible.

(1) Direct Potential Measurement

Placing the Operating Mode switch in the DIRECT MEAS. ONLY mode sets the instrument up for a direct potential measurement. In this mode the meter directly indicates the potential of the Working Electrode with respect to the Reference Electrode (Working Electrode is at ground; the Reference Electrode is off ground as determined by the cell and electrode characteristics). The potential at the ELECTROMETER MONITOR connector will be that of the Reference Electrode with respect to ground. As in CONTROL E operation, the Selector must be in the EXT. CELL position to obtain a meter indication. The Meter Function switch should be set to POTENTIAL. The Counter Electrode circuit is open in this mode and there is no cell current.

(2) Null Potential Measurement

The NULL mode allows high resolution measurements of electrode potentials to be made. In this mode, the electrodes are connected as before (except for the Counter Electrode, which is open), but the meter indicates the difference between the electrode potential and the set Applied Potential. Specifically, when the output of the "active" set of controls is adjusted as required to achieve meter null, the control setting will directly indicate the potential of the Working Electrode with respect to the Reference Electrode. (As in the other modes, the Working Electrode is actually at ground potential and the Reference Electrode is off ground as determined by the cell and electrode characteristics.) Using this method allows potentials to be read to within one millivolt.

4.4 MODEL 173 WITH DIGITAL READOUT

If requested at the time of purchase, a 3½ digit Nixie* display will be supplied. This display provides direct numerical readout of the Model 173's output and the corresponding digital data is available at a rear-panel connector. This data is suited to computer processing.

In reading the display, the numerals and polarity correspond directly to the monitored voltage or current. On any "1" range, 1 μ A, 10 μ A, 100 μ A, etc., the display indicates 1.000 full scale. On any "2" range, the maximum possible display is 1.999. With a higher output, the display becomes 1 BLANK, that is, the "1" remains lighted, but the

^{*}Nixie is a trademark of Burroughs Corporation.

decimal point and all following digits go dark. On any "5" range, full-scale output gives a display indication of 5.00, that is, the decimal point is shifted one place to the right.

The information displayed on the digital panel meter, along with the exponent data, is provided in BCD form at rear-panel connector J23. The tables following give the signal/pin assignments for this data. Positive logic is employed. A "1" is $\pm 3.5 \text{ V} \pm 1 \text{ V}$ and a "0" is $0.2 \text{ V} \pm 0.2 \text{ V}$. All digital output signals are capable of sinking 5 mA at the lower level and of sourcing 0.1 mA at the upper logic level.

Occasion may arise where it is advantageous to trigger externally, such as when it is necessary to operate in conjunction with certain peripheral digital processing equipment. NOTE: For operation with the Model 131 system, internal triggering is employed. Considerations that govern external triggering are that the internal trigger circuits must be inhibited and that the proper external trigger be applied. Internal triggering is inhibited by applying ground to pin 23 of the digital output connector. The external trigger should be applied to pin 20. The required signal is a logic one that goes to logic zero for at least one and a half microseconds (but for less than two milliseconds). The unit resets on the negative-going transition; conversion commences on the positive-going transition. The maximum allowable external trigger rate is 60

NOTE: Early Model 173's equipped for digital readout were furnished with an Analogic Model 2510 Digital Voltmeter. Current production units are supplied with an Analogic Model 2532 Digital Voltmeter. Although similar, these meter do differ in some respects.

From an operating point of view, the primary difference is that the current voltmeter does not have a "+" sign. If no polarity is indicated, the reading is presumed to be positive. A "-" sign is provided to indicate negative readings.

The other changes involve pin assignments and signal levels. The old and current meters are compared in the following table.

Model 2510 (Old Meter)		Model 2532 (New Meter)	
Pin	Function	Pin	Function
DJ-1 (L)	+ 4.5 V	DJ-2 (M)	+ 4.5 V
DJ-2 (N)	+ Overload Gate	DJ-1 (L)	+ Overload Gate
DJ-2 (M)	+ 4.5 V	DJ-2 (N)	- Overload Gate
DJ-2 (S)	- Transition Trig	DJ-2 (S)	+ Transition Trig

The "Transition Trigger" signal is actually the External Trigger Input at pin 20 of the Digital Inter-

face connector. In units equipped with the Model 2510 Digital Panel meter, conversion commences on the trailing edge (– transition) of a logic 1 applied to this input. In current units, those equipped with the Model 2532 panel meter, conversion commences on the leading edge (+ transition) of the applied logic one (unit resets on preceding negative transition; logic 0 to be longer than 1.5 μs but less than 2 ms).

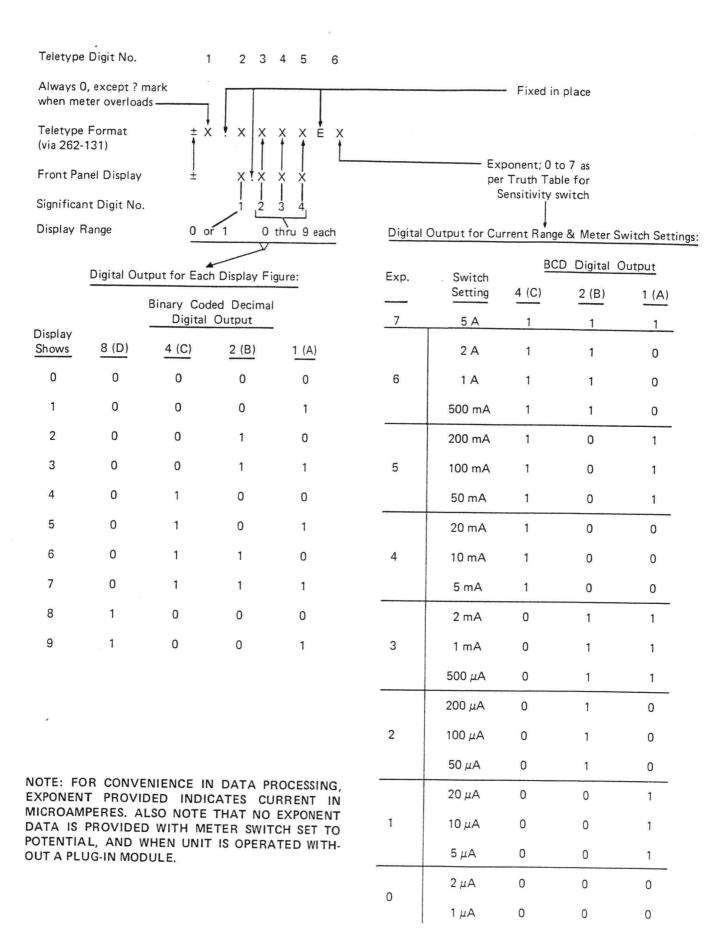
1	Polarity (logic 1 = +).	
2	+4 V dc (not used in M131 System).	
3, 4, 28, 29	Overrange Output (all four pins tied together inside M173). M131 system prints question mark in most significant place* if the digital panel meter is driven into overrange.	
5	2º of most significant digit. 2¹ of most significant digit. This line is always grounded inside the M173. 2² of most significant digit. This line is always grounded inside the M173. 2³ of most significant digit. This line is always grounded inside the M173.	
7	2° of second digit.	
32	2' of second digit.	
8	2° of second digit.	
33	2° of second digit.	
9	2° of third digit.	
34	2¹ of third digit.	
10	2² of third digit.	
35	2³ of third digit.	
11	2° of fourth digit.	
36	2¹ of fourth digit.	
12	2² of fourth digit.	
17	2³ of fourth digit.	
27	ground.	
23	Internal Trigger Inhibit. Logic 0 at this point inhibits internal triggering. M131 BUSY applied to this pin.	
20		
19	CONVERSION COMPLETE (complement of signal at pin 18).	
26	2° of exponent digit**. 2° of exponent digit**. 2° of exponent digit**.	

Table IV-2. DIGITAL OUTPUT PINS AND SIGNALS

NOTE: A cable suitable for interfacing the Model 173 digital output with the Model 262 Teleprinter Interface module of the Model 131 system is available. The cable part number is 6020-0023-06. The drawing number for this cable is 5401-C-ESA.

^{*}Most significant digit of M173 digital display is printed in second most significant digit place of M131 system printout. The most significant place in the printout is reserved for a digital panel meter overrange indication. When overrange of the panel meter occurs, a question mark is printed in the most significant place. Otherwise a zero.

zero.
**Exponent only provided if unit is equipped with a Model 176/D, a Model 179, or a Model 376.



4.5 BIBLIOGRAPHY

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SECTION V TROUBLESHOOTING

WARNING!

THESE SERVICE INSTRUCTIONS ARE FOR USE BY QUALIFIED PERSONNEL ONLY. TO AVOID ELECTRIC SHOCK, DO NOT PERFORM ANY SERVICING OTHER THAN THAT CONTAINED IN THE INSTRUCTIONS UNLESS YOU ARE QUALIFIED TO DO SO. POTENTIALLY LETHAL VOLTAGES ARE PRESENT INSIDE THIS APPARATUS.

ANY ADJUSTMENT, MAINTENANCE OR REPAIR OF THE OPENED APPARATUS UNDER VOLTAGE SHALL BE AVOIDED AS FAR AS POSSIBLE AND, IF INEVITABLE, SHALL BE CARRIED OUT ONLY BY A SKILLED PERSON WHO IS AWARE OF THE HAZARD INVOLVED.

5.1 INTRODUCTION

When a problem is first noticed, the first task is to determine whether it has its origin in the M173 mainframe itself or outside the M173. Many problems result from cabling or cell failings of one kind or another. Section III contains some initial checks that can be used to check out the basic functioning of the mainframe. If the indicated behavior is observed, one can be reasonably sure that the instrument is functioning correctly, and that the problem has its origin in the other system components. The following paragraphs discuss some possible external problems, and then go on to discuss specific mainframe troubleshooting procedures.

5.2 EXTERNAL PROBLEMS

- (1) OSCILLATION: One of the most commonly encountered problems is that of high frequency oscillations. Such oscillations can be observed by monitoring the rear-panel S/A OUT connector with an oscilloscope, or by monitoring the ELECTROMETER MONITOR connector. This oscillation is usually the result of phase shifts introduced by stray capacitance and the high impedance of the Reference Electrode. Some possible remedies include:
 - (a) Check the Reference Electrode itself. Be sure that it is properly filled, that it is immersed, and that the filling hole is exposed. Also check that the Reference Electrode isn't clogged. If clogging is suspected, disassemble the electrode, clean it, and fill it with fresh saturated KCI. NOTE: One way of checking for clogging is to immerse the electrode in a beaker of water. If an electrode is "leaking" at a sufficiently high rate, a "thread" of KCI will soon become visible. Unfortunately, the

- leakage rate of electrodes that are working perfectly well can be too low to allow a visible thread to be observed.
- (b) Check the cable connections. In particular, be sure that the lead length from the Reference Electrode to the Electrometer is no longer than necessary. The lead coming off the Reference Electrodes provided by EG&G Princeton Applied Research Corporation will plug directly into the white-tipped jack on the face of the Electrometer Probe. Take care not to unduly lengthen this lead by means of an unnecessary clip lead. Also, do not allow this lead to come near any clamps of other associated metalware that is at ground potential. The Reference Electrode, and the lead from the Reference Electrode to the Electrometer Probe must be kept as far from any grounded objects as possible. If a 178/41 Filter is used, keep its leads short as well, and away from grounded objects.
- (2) OVERLOAD LIGHT ON: The most common cause of the Overload light glowing is incorrect cabling. Check to be sure the red clip is connected to the Counter Electrode, and that the electrode is immersed. Similarly check the Working Electrode connection (green clip), and be sure that the electrode is immersed. If the Working Electrode is an HMDE, be sure that the drop hasn't been accidentally dislodged. Check that the black alligator clip (ground) isn't accidentally shorting to another circuit element, in particular the Working Electrode or Counter Electrode. If the problem is excessive 60 Hz pickup, it may help to shield the entire assembly, grounding the shield (screen) with the black clip.

5.3 INTERNAL CHECKS

If it has been determined that there is an internal malfunction, the following procedure should be followed to localize the problem to a particular circuit in the instrument. This will require removal of the top cover which is held in place by two screws under the "overhang" at the rear of the instrument.

(1) Unregulated Voltage Checks

Measure the unregulated voltages at the rectifier board fuses as shown in Figure V-1. Any voltage measuring device with sufficient range can be used for this measurement. If

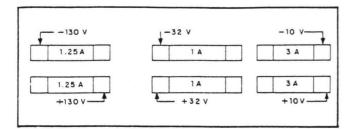


Figure V-1. RECTIFIER BOARD (TOP VIEW)

any of the above voltages are zero, the associated fuse is probably blown and should be replaced.

(2) Regulated Voltage Checks

NOTE: The +24 V supply is the key supply from which all the other supplies are regulated. If the +24 V supply is not working, none of the supplies will be working.

If it has been determined that the unregulated voltages are as specified, the regulated voltage should be checked, using the test points located along the top edge of the power supply board. The voltages should be measured with respect to chassis ground.

Red Test Point: $+24.4 \text{ V} \pm .1 \text{ V}$ Yellow Test Point: $-24.4 \text{ V} \pm .1 \text{ V}$ Violet Test Point: +105 V (+102 V to +108 V)White Test Point: -105 V (-102 V to -108 V)

If any of these voltages are not as specified, proceed to Subsection 5.6 to determine the exact malfunction. If the voltages are as specified, proceed to the particular section which describes the malfunction.

5.4 A OR B POTENTIAL CONTROL MALFUNCTION

A malfunction in this section will show one of the following symptoms:

- (1) Either the A or B pushbutton will not light.
- (2) One or both polarities of either the A or B Potential Control will be inoperative.
- (3) One of the External Triggering functions will be inoperative.
- (4) The ZERO I pushbutton will not light when pushed with the Operating Mode switch set to CONTROL I.

Any of the above symptoms indicates a failure on the Control Logic Board which is located nearest the front panel. Detailed troubleshooting of this board is beyond the scope of this manual. Defective boards should be returned to the factory for replacement.

5.5 SUMMING AMPLIFIER MAL-FUNCTION

A malfunction in this section will show one of the following symptoms:

- (1) Overload light always on.
- (2) Thermal overload cutting in.
- (3) Fuses on Rectifier Board blow when power is turned on.
- (4) Summing Amplifier output shows large dc offset.

To troubleshoot a Summing Amplifier, the power stage heat sink should be disconnected and a heat-sink jumper installed in its place. With the cover off and observing the instrument from the front, the operator will observe a cable extending from the heat-sink assembly at the right rear of the instrument to a chassis connector just in front of the heat-sink assembly. The power stage heat sink is disconnected (Power OFF) by unplugging this cable from the chassis connector. A so-called "heat-sink jumper" is then plugged into the chassis connector in place of the connector at the end of the cable. A heat-sink jumper is constructed by taking the same type connector as that at the end of the cable (AMPHENOL 57-30240) and adding a jumper and two resistors to it. A jumper is connected from pin 21 to pin 22. One 100 ohm half-watt resistor is connected between pins 17 and 18. Another is connected between pins 13 and 14. The completed heat-sink jumper can then be plugged into the chassis connector and the power turned back on. The procedure follows.

- Connect a scope or voltmeter to the rearpanel connector marked S/A OUT.
- (2) Set the front-panel switches as follows.

A and B Potentials: OFF Operating Mode: CONTR. E Cell Selector: OFF External Signal Inputs: OFF

- (3) Turn the power on and observe the overload light and the scope or meter.
- (4) If the overload light is out and the scope or meter reads zero, then switch either the A or B Potential Control to +1 V and determine that the voltage at the S/A OUT connector is -1 V.

- (5) If step (4) above gives a normal output, the problem probably is a bad transistor on the power stage heat sink. In this case each transistor on the heat sink should be checked.
- (6) If step (4) above does not give a normal output and the power supply voltages are normal (see Subsection 5.3), then the trouble is most likely on the Summing Amplifier Board itself. Return the board to the factory for service.

5.6 POWER SUPPLY MALFUNCTION

A malfunction in this section will show one of the following symptoms:

- (1) Overload light always on.
- (2) One or both panel meter lights out.
- (3) One or all pushbutton lights out.
- (4) Large dc offset on the Summing Amplifier output.
- (5) Low or high voltage readings on the unregulated power supplies.

POWER SUPPLY TROUBLESHOOTING PROCEDURE

If the regulated voltage is very low, there may be heavier than normal loading on the supply caused by a malfunction elsewhere in the unit. To determine if this is the case, the steps below should be followed:

- (1) Turn the power off and unplug the unit.
- (2) Remove the Control Logic Board and the Summing Amplifier Board. Then unplug the Power Stage heat sink, the Model 176, and the Model 178, and install a heat sink jumper in place of the Power Stage heat sink.

NOTE: With the top cover of the instrument removed, and facing the instrument from the front, the operator will observe that the heat sink and fan assembly is located at the rightrear corner of the instrument. Normally an aluminum cover bearing a HIGH VOLTAGE warning prevents direct access to this assembly. A cable extends from the assembly to a connector located on the chassis. This connector is just in front of the heat-sink assembly and just behind the rear-most circuit board. The specific requirements of this step are that the cable connector be unplugged from the chassis connector and a "heatsink jumper" be plugged into the chassis connector in place of the cable connector. See Subsection 5.5 for instructions on how to construct a heat-sink jumper.

- (3) Turn the power on and measure the voltage at the RED test point near the top edge of the Power Supply Board. This test point should measure + 24.4 V ± 1 V. If this voltage is wrong, refer to the Power Supply Schematic (page VII-7) and troubleshoot the + 24 V supply first.
- (4) If the correct voltage is measured at the red test point, then proceed to the - 24 V supply. This is the YELLOW test point on the Power Supply Board. If this voltage is wrong, refer to the Power Supply Schematic (page VII-7) and troubleshoot the - 24 V regulator.

Once both low voltage power supplies are working properly, the high voltage can be checked. Note that the two high voltage pass transistors are located on the Power Stage heat sink, and are not in use when the heat-sink jumper is used. These transistors should be checked separately, especially if either of the 1.25 A fuses on the Rectifier Board were blown.

A possibility not discussed above is that of a thermal overload switch (S13 and S14 located on the heat-sink assembly) failing. To check these switches, simply disconnect the heat-sink cable from the chassis connector as described previously. Then measure continuity between pins 21 and 22 of the *cable connector* with an ohmmeter. An open indicates that either S13 or S14 is defective. A schematic of the heat-sink wiring is located on page VII-12.

POWER SUPPLY LOADING

If the power supply voltages returned to normal when the boards were removed, the following procedure can be used to determine which board is loading the power supplies.

- (1) Turn the power off and install the Control Logic Board.
- (2) Turn the power on and note that the A potential pushbutton light comes on and the power supply voltages remain normal.
- (3) Turn the power off and install the Summing Amplifier Board with the heat sink jumper still in place.
- (4) Turn the power on and observe the overload light and check the S/A OUT connector for approximately zero volts. Also check that the power supply voltages remain normal.
- (5) Continue with the Power Stage heat sink, then the plug-in module (if one is used), and last the Model 178.

With this procedure one should be able to determine which board or plug-in is causing the problem.

SECTION VI CALIBRATION PROCEDURE

WARNING!

THESE SERVICE INSTRUCTIONS ARE FOR USE BY QUALIFIED PERSONNEL ONLY. TO AVOID ELECTRIC SHOCK, DO NOT PERFORM ANY SERVICING OTHER THAN THAT CONTAINED IN THE INSTRUCTIONS UNLESS YOU ARE QUALIFIED TO DO SO. POTENTIALLY LETHAL VOLTAGES ARE PRESENT INSIDE THIS APPARATUS.

ANY ADJUSTMENT, MAINTENANCE OR REPAIR OF THE OPENED APPARATUS UNDER VOLTAGE SHALL BE AVOIDED AS FAR AS POSSIBLE AND, IF INEVITABLE, SHALL BE CARRIED OUT ONLY BY A SKILLED PERSON WHO IS AWARE OF THE HAZARD INVOLVED.

6.1 EQUIPMENT REQUIRED

- (1) Oscilloscope, Tektronix Model 561B or equivalent.
- (2) DC Null Voltmeter, H.P. Model 419A or equivalent.
- (3) Digital Voltmeter, Fairchild Model 7000 or equivalent.
- (4) Model 178 Electrometer Probe.
- (5) External Cell Cable (Model 173).
- (6) Differential Voltmeter, H.P. 3420 or equivalent.

6.2 POWER SUPPLY ADJUSTMENT

NOTE: Instrument must be turned on with all covers installed for a minimum of one hour.

- (1) Slide the top cover back. Then monitor TP201 (red test point) with the digital voltmeter and set R202 (+ 24 V ADJ.) for a DVM indication of + 24.40 V*.
- (2) Transfer the DVM to TP203 (yellow) and set R221 (-24 V ADJ.) for a DVM indication of -24.40 V*.
- (3) Check the voltage at TP202 (violet test point). The voltage should be in the range of + 106 V to + 112 V.
- (4) Check the voltage at TP204 (white test point). The voltage should be in the range of - 106 V to - 112 V.

6.3 SUMMING AMPLIFIER ZERO ADJUSTMENT

- (1) Set the "A" and "B" potentials to OFF and center the rear panel S/A Zero trim-adjustment (R3).
- (2) Connect the dc nullmeter to the S/A OUT rear panel connector.
- (3) Adjust R307 (SUMMING AMP. ZERO ADJ.) for zero \pm 50 μ V.
- (4) Adjust S/A Zero (R1, rear panel) and check its effect on the voltage at the S/A OUT connector. The range should be about $\pm 250~\mu V$. Leave R1 set for zero $\pm 10~\mu V$.

6.4 A AND B POTENTIAL RANGE CALIBRATION

- (1) Connect the Differential Voltmeter (HP3420) to the S/A OUT connector (rear panel), set the A potential to +4.000 V, and adjust R11 (+ A POTENTIAL ADJ.) for a reading of -4.000 V at the HP3420.
- (2) Change the A potential setting to -4.000 V and adjust R12 (-A POTENTIAL ADJ.) for a reading of +4.000 V.
- (3) Change the A potential setting to -0.900 V and set the A ADJ. potentiometer (located on the resistor summing board) for a reading of +0.900 V.
- (4) Transfer to B potential control. Then set the B potential to +4.000 V and adjust R10 (+B POTENTIAL ADJ.) for a reading of -4.000 V.
- (5) Change the B potential setting to -4.000 V and adjust R13 (-B POTENTIAL ADJ.) for a reading of +4.000 V.
- (6) Change the B potential setting to -0.900 V and set the B ADJ. potentiometer (located on the resistor summing board) for a reading of +0.900 V.

6.5 SUMMING AMPLIFIER CURRENT LIMIT

- (1) Adjust R348 and R343 fully clockwise.
- (2) Set up an A potential of -1.000 V and set the current range to 1 A. The Electrometer Probe should be connected to an external resistor as shown in Figure III-1 (page III-2), except

^{*}This is not a printing error. The correct voltage is 24.40 V and not 24.00 V.

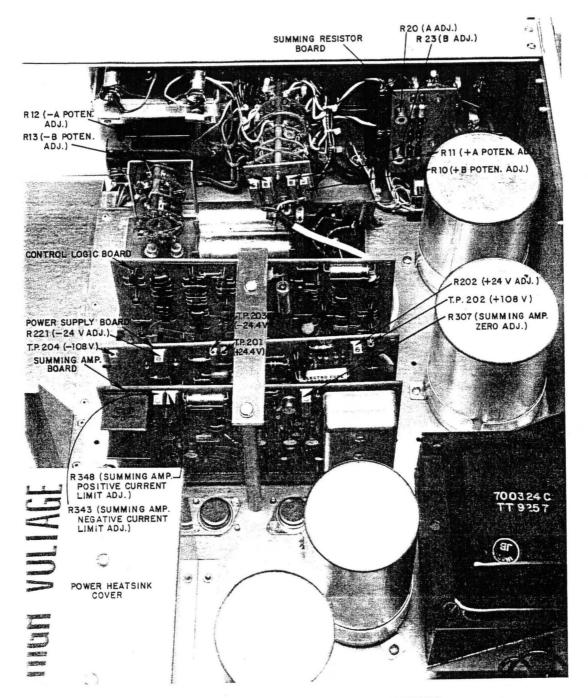


Figure VI-1. LOCATION OF MODEL 173 ADJUSTMENTS AND TEST POINTS

that in this case a 1 Ω ± 1% resistor rated at two watts is used instead of the 1 k Ω resistor shown in the figure.

- (3) Set the Cell Selector to EXT. CELL and adjust R348 (SUMMING AMP. + CURRENT LIMIT ADJ.) until the overload light just comes on. Monitor both the S/A output and the Model 176 output to assure stable operation.
- (4) Switch to + 1.000 V and adjust R343 (SUM-

MING AMP. – CURRENT LIMIT ADJ.) until the overload light just comes on.

6.6 MODEL 178 ELECTROMETER PROBE ZERO ADJUSTMENT

NOTE: The Electrometer Zero adjustment is located behind the white nylon screw on the front of the probe.

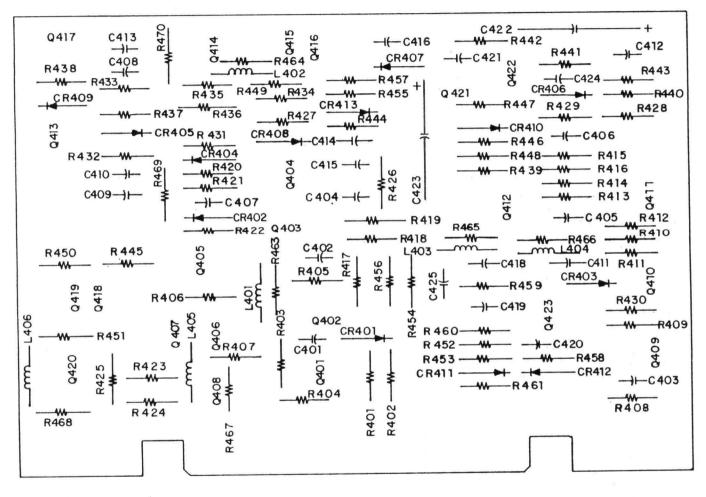
- (1) With the Cell Selector switch to OFF, connect the Electrometer input (white tip jack) to ground (black lead of the external cell cable).
- (2) Connect a dc voltmeter to the front panel

Electrometer Monitor connector.

- (3) Set the Electrometer Zero adjustment for a reading of zero \pm 10 μ V.
- (4) Replace the nylon screw.

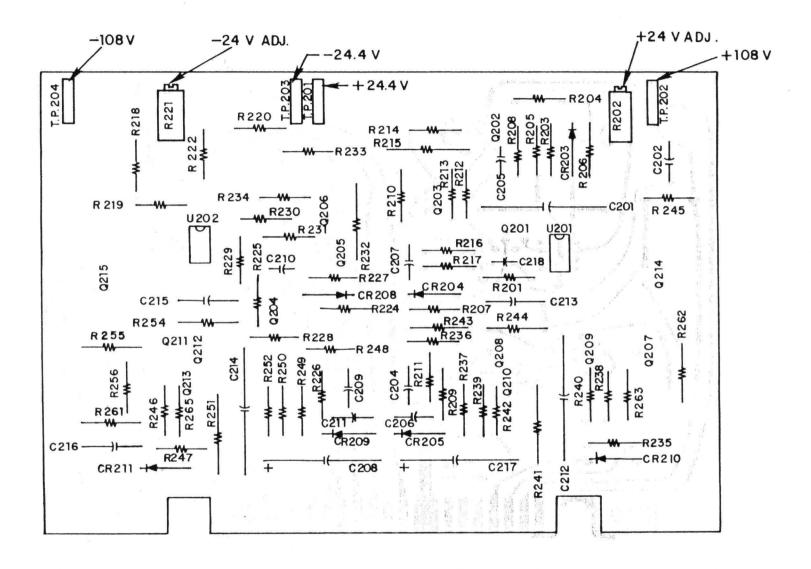
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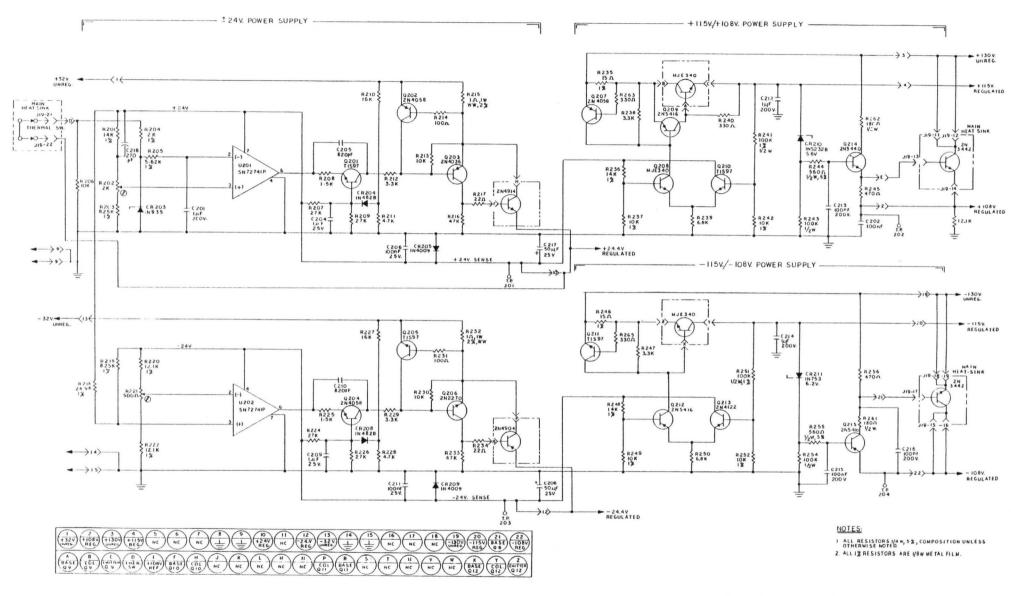


SYMBOLIZATION MODEL 173 CONTROL LOGIC BOARD

(

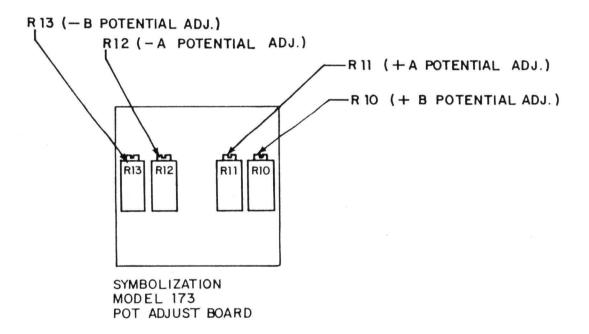


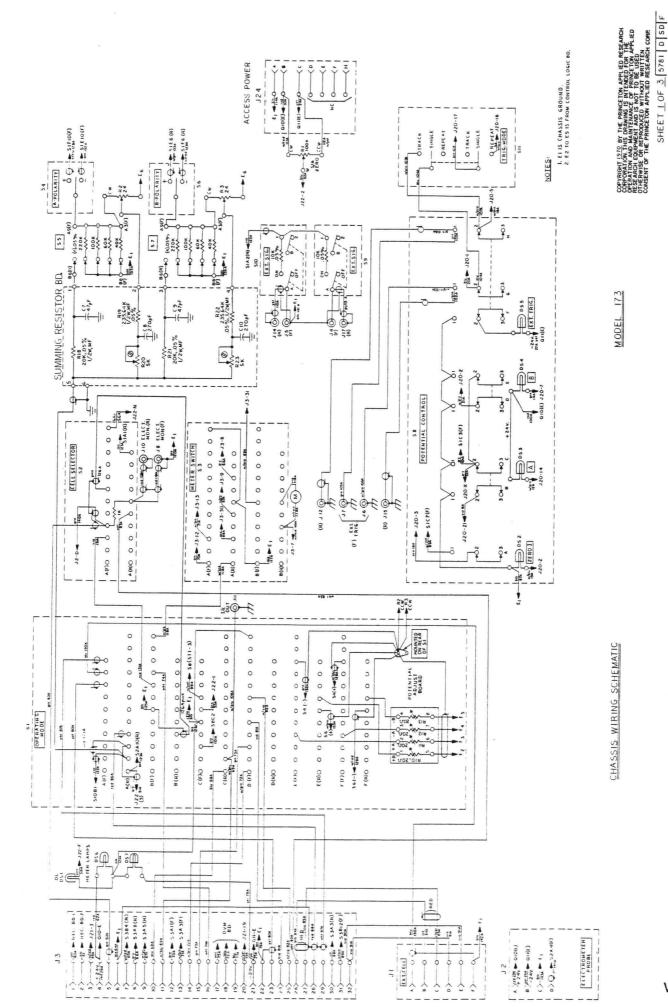
SYMBOLIZATION MODEL 173 POWER SUPPLY BOARD

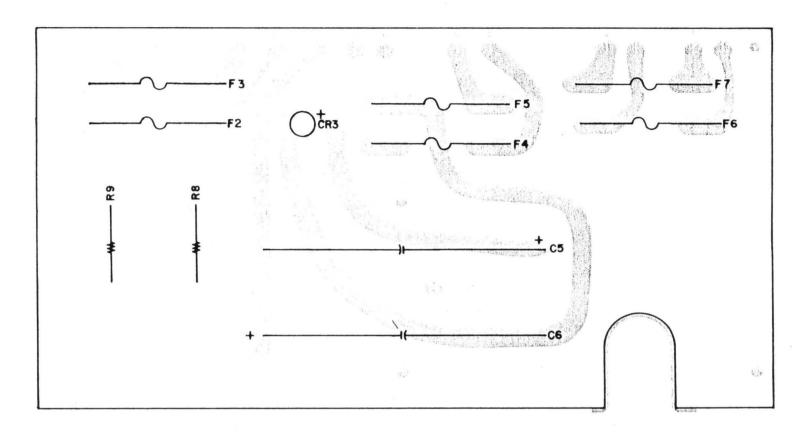


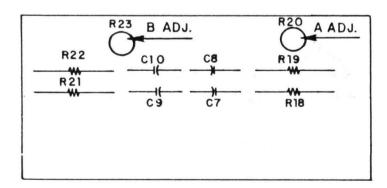
POWER SUPPLY BOARD SUB ASSEMBLY 1730-22-0009S MODEL 173

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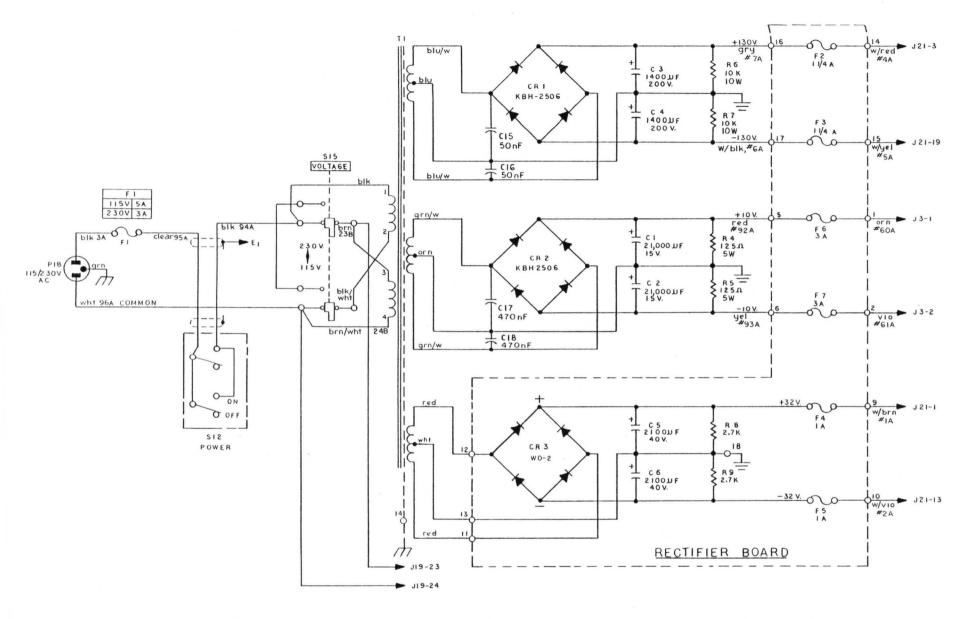






SYMBOLIZATION MODEL 173 SUMMING RESISTOR BOARD

SYMBOLIZATION MODEL 173 RECTIFIER BOARD



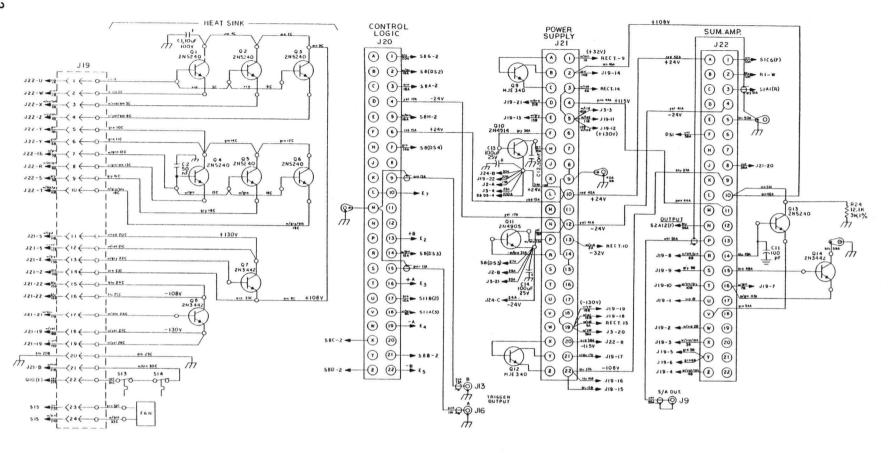
CHASSIS WIRING DIAGRAM MODEL 173

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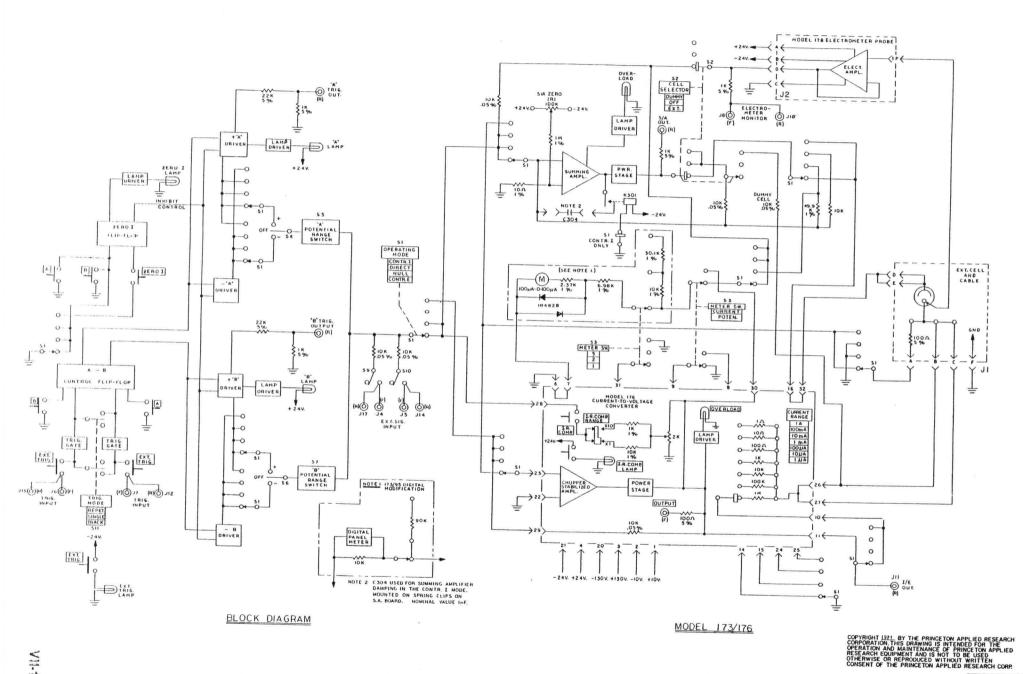


CHASSIS WIRING DIAGRAM

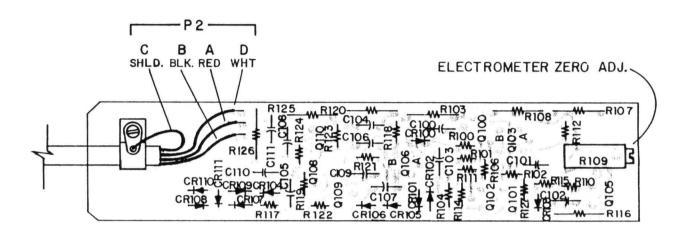
MODEL 173

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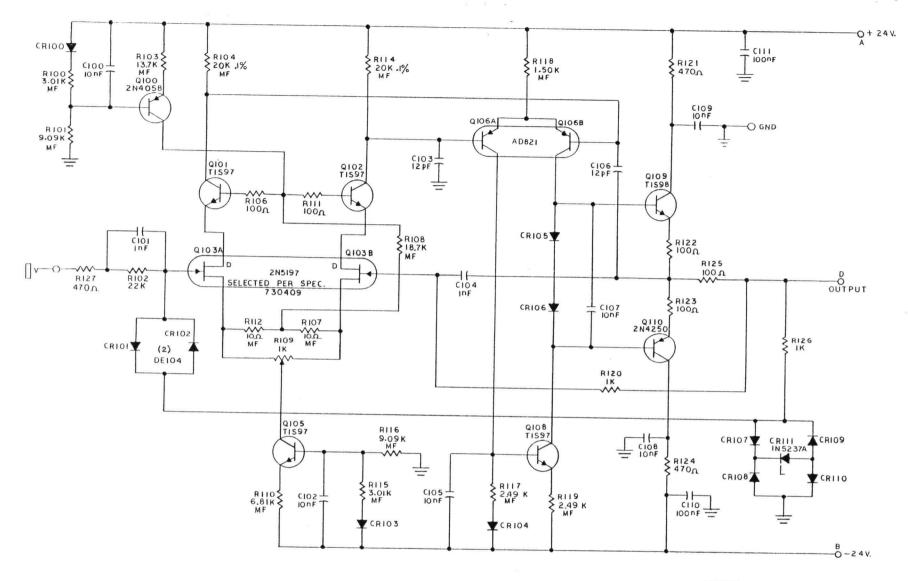
SHEET 2 OF 3 5781 D SD



6414 D SD



SYMBOLIZATION
MODEL 178
ELECTROMETER PROBE BOARD



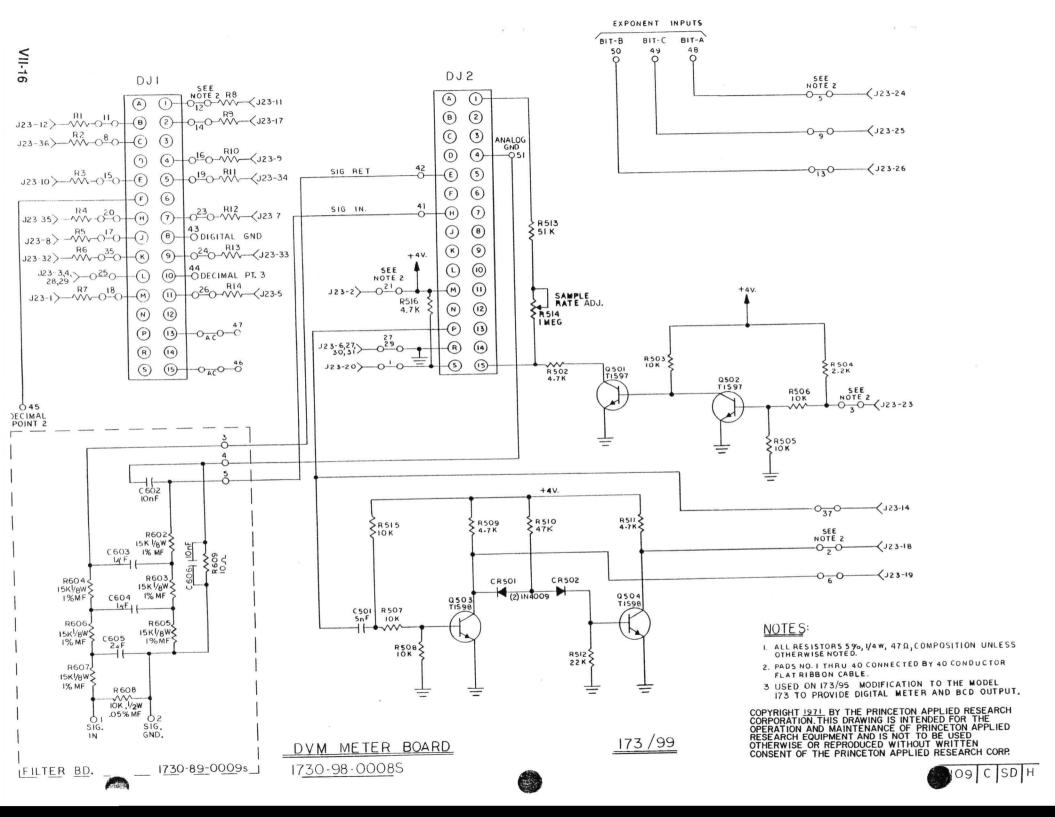
ELECTROMETER PROBE

MODEL 178

NOTES:

- I. ALL RESISTORS \$4\$,5\$, COMPOSITION UNLESS OTHERWISE NOTED.
- 2. MFINDICATES V8W,1%, METAL FILM RESISTOR UNLESS OTHERWISE NOTED.
- 3. ALL DIODES IN 4828 UNLESS OTHERWISE NOTED.

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APPENDIX A

MODEL 380 CONSTANT POTENTIAL COULOMETRY SYSTEM

INTRODUCTION

The Model 380 Controlled Potential Coulometry System provides an instrument package and cell system suitable for performing rapid, accurate CPC (controlled potential coulometry) analyses. Included in the system are a Model 173 Potentiostat/Galvanostat, a Model 179 Digital Coulometer, and a complete 377A Cell System.

The Model 173 gives the system a compliance voltage range of $\pm 100~V$ at currents as high as 1 A. There is provision for transient-free switching between two independent potential/current control channels. This feature gives the system the capability of performing multiple coulometric analyses at different potentials, or even for examining both reduction and oxidation processes at different potentials. Transfer from one control channel to the other can be accomplished by means of front-panel controls or by remotely derived control signals. The Model 178 Electrometer Probe furnished with the Model 173 allows extreme high-impedance monitoring of the Reference Electrode. The probe is located at the end of a long cable so that it can be readily positioned close to the Reference Electrode, thereby minimizing electrical pickup and the possibility of oscillation due to stray cable capacitance.

The Model 179 Digital Coulometer plugs into the Model 173 to form a state-of-the-art instrument package for making precision coulometric measurements at either a controlled potential or controlled current. Quantities from ten picoequivalents to ten equivalents can be measured coulometrically with an absolute accuracy of better than ±0.1% of full scale. Although a typical analysis can generally be completed in a few minutes, the drift of the coulometer is so low that experiments lasting hours or even days can be accommodated, if necessary. A four-digit plus polarity and exponent digital display continuously indicates the accumulated charge. There is provision for automatic subtraction of any background current. Furthermore, the displayed charge accumulation is provided in BCD format at a front-panel Digital Output connector to facilitate automatic data recording or computer processing of the signal, if desired.

The Model 377A satisfies the CPC requirement for a special cell arrangement, one capable of bringing all of the sample into contact with the working electrode in the minimum possible time. The Model 377A is designed with this goal in mind with the result that complete electrolyses are typically completed in 6-8 minutes. In addition to providing all of the glassware and hardware items required for controlled potential coulometry at a mercury working electrode, the system includes a stand kit, a synchronous stirring motor, and a noise filter. For electrolyses requiring a platinum working electrode, an optional Model K0027 Platinum Electrode can be easily installed. The Model 377A is also designed with a mind to operator convenience. All the electrodes and glassware items are affixed to the cell top.

The cell bottom can be either secured to the cell top or removed from it in seconds, allowing samples to be readily changed without having to disassemble the entire cell system.

Thus, the Model 380 Controlled Potential Coulometry System brings together all the components necessary to easily perform rapid, precision coulometric analyses. The operator has only to assemble the system, provide the working electrode (mercury or K0027 platinum electrode), and add the sample to use this highly accurate and precision analytical technique. No standard solutions, calibration curves, end-point detectors, or other peripheral apparatus is required. The current-time integral of the completed electrolysis gives the number of equivalents in the sample directly, with a typical analysis taking but a few minutes to complete.

SYSTEM COMPONENTS

- (1) Model 173 Potentiostat/Galvanostat with Model 178 Electrometer Probe and a cell cable terminated in three alligator clips.
- (2) Model 179 Digital Coulometer (plugs into Model 173).
- (3) Model 377A Cell System containing:
 - 1 Model K0028 Coulometry Stand Kit
 - 1 Model 377 Synchronous Stirring Motor
 - 1 Accessory 178/41 Noise Filter
 - 1 Model K0026 Coulometry Cell Kit, containing:
 - 3 Model G0062 Cell Bottoms
 - 1 Model MP0447 Cell Bottom Retaining Ring
 - 2 Model G0063 Mercury Stirring Rods
 - 1 Model G0065 Outgassing Tube
 - 1 Model G0066 Counter Electrode Bridge Tube
 - 1 Model G0067 Reference Electrode Bridge Tube
 - 1 Model K0029 Cell Top Kit
 - 1 Model K0030 Platinum Counter Electrode Kit
 - 2 Cell Support Rods
 - 1 Model S0026 Sample Port Stopper
 - 1 Model K77 Saturated Calomel Ref. Electrode
 - 5 Model G0070 replacement Vycor frits
- (4) Three instructions manuals, one for the Model 173, one for the Model 179, and one for the 377A Cell System.
- (5) Note that Working Electrodes are not supplied. A mercury pool is most often used as a working electrode for electrolyses involving a reduction process. Oxidations are normally carried out with a platinum working electrode. A suitable platinum electrode (Model K0027) is available as an option.

R Vycor is a registered trademark of Corning Glass Corporation.

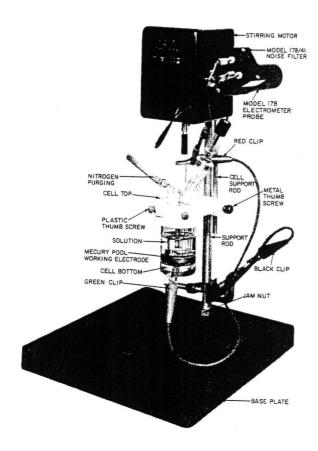


Figure 1. MODEL 377-1 MERCURY-ELECTRODE COULOMETRIC CELL

SYSTEM ASSEMBLY (Figure 1)

- (1) Refer to the Model 377A Instruction Manual and assemble the Model 377A Cell System as described therein.
- (2) Plug the Model 179 Digital Coulometer into the Model 173.
- (3) Connect the Cell Cable (terminates in three alligator clips) and the Electrometer cable (terminates in Electrometer probe) to the proper Model 173 frontpanel connectors.
- (4) At the cell Assembly, the Electrometer Probe should be mounted in the clamp at the side of the Stirrer Motor. Then the Model 178/41 Noise Filter should be attached to the Electrometer Probe (see Figure I-1 of the 377A manual).
- (5) After checking to be sure that the Model 173 Cell Selector is set to OFF, connect the red clip at the end of the cell cable to the Counter Electrode. Then connect the green clip to the Working Electrode (feedthrough at the bottom of the cell if mercury electrode; proper cell top connection if platinum

electrode). The black clip (ground) is unused. However, it may be advisable to connect it to a cell-support rod just to get out of the way so it won't short against one of the active circuit elements.

- (6) Plug the Reference Electrode lead directly into the white jack on the Model 178 Electrometer Probe. Then extend a short lead from the connection just made to one of the pin jacks on the Model 178/41 Noise Filter. Run a second short lead from the other Model 178/41 Noise Filter pin jack to the counter electrode. In other words, there will be two connections at the counter electrode (red clip of cell cable and lead from one pin jack of noise filter) and two connections at the Model 178 Electrometer Probe (Reference Electrode lead and lead from other pin jack of noise filter).
- (7) Connect a source of dry nitrogen from which trace oxygen has been removed to the outgassing tube. This gas will purge the sample of dissolved oxygen and so prevent interfering oxygen reactions.

This completes the system assembly. Before attempting to operate the system, the customer is well advised to read Section III of both the Model 377A Instruction Manual and the Model 179 Instruction Manual. It is also advisable to read the following discussion of the Vycor-tipped Bridge Tubes.

HANDLING INSTRUCTIONS FOR GLASS-WARE INCORPORATING VYCOR TIPS

Both bridge tubes incorporate an unfired Vycor tip designed specifically to provide ultra low leakage rates with minimum IR drop through the tip. This arrangement eliminates complications arising from poisoning of test solutions by electrode filling solutions or by unwanted species produced at the counter electrode.

The Vycor tip is deliberately installed in the dry state. ONCE THE TIP IS WET, IT MUST BE KEPT WET. This means that when not in use, the tube must be stored in a solution-ideally a solution that closely approximates the solutions employed in the electrochemical experiment. If the tip is allowed to remain dry for more than a few minutes once it has been wet, it will crack and have to be replaced. Also, the tip might crack if it is exposed to drastically different environments. For example, one should not take a bridge tube filled with an organic solvent and immerse it in an aqueous solution, or vice versa. If it should be necessary to use the bridge tube in different environments, e.g., organic solvents and aqueous solutions, it may be possible to prevent the tip from cracking by equilibrating the tip (inside and out) with a mixture (or mixtures) of the two environments. Alternatively, two sets of bridge tubes should be used-one for each environment.

If a Vycor tip should be damaged, it can be replaced as follows. First slice off the old Teflon® sleeve with a razor

[®] Teflon is a registered trademark of E. I. DuPont Co.

blade and discard the sleeve and damaged disc. Wash and dry the bridge tube. Then slip a new length of heat-shrink Teflon tubing onto the bridge tube. Stand the bridge tube on the new Vycor disc (DO NOT WET THE TIP AND AVOID FINGER CONTACT WITH IT). Heat the Teflon tubing on all sides with a hot-air stream from a heat gun. A

direct flame should not be used because it will char the Teflon. Allow the tubing to shrink over the disc. Be sure to let the tubing cool to room temperature before lifting the assembly. Extra tips and Teflon sleeves are provided with the system. More can be purchased from Princeton Applied Research Corporation if necessary.

APPENDIX B MODELS 331-1, 331-2, AND 331-3 CORROSION MEASUREMENT SYSTEMS

INTRODUCTION

These systems have been designed to greatly simplify corrosion investigations. At the heart of all three systems is the PARC Model 173 Potentiostat/Galvanostat equipped with a Model 376 Logarithmic Converter. This combination gives the corrosion investigator a powerful tool that is easy to operate. The potentiostat features an output voltage range of ±100 V at 1 A, with corrosion potential measurements accurate to ±1 mV. The bipolar logarithmic converter provides current measurements over five decades with an accuracy of ±0.2%, with provision for displaying the current either linearly or logarithmically on the Model 173 panel meter. These systems incorporate a voltage programmer to control the potentiostat/galvanostat. There is no need for continuous operator attention or tedious point-by-point measurements, even for log current-potential plots. Once started, the entire experiment is conducted automatically.

Measurements that can be performed with these systems include automatic potentiodynamic anodic polarization plots, Tafel plots, linear polarization, corrosion potentials, and galvanic corrosion. Other techniques such as galvanostatic, chronoamperometric, and cyclic voltammetric measurements are easily performed to assist in characterizing electrode materials.

The Model 331-3 System includes a Model 175 Universal Programmer and a Model K0047 Corrosion Cell Kit for maximum utility and flexibility. The Model K0047 Corrosion Cell Kit contains the necessary cell, glassware, and hardware for assembling a corrosion investigation system ideally suited to operation with a Model 173 controlled by a Model 175. The Model 175 Programmer features bipolar operation with as many as four inflection points on a single output waveform and both single cycle or continuous operation. Either pulse or ramp operation can be selected. In sweep operation, the amplitude and slope are independently adjustable. In pulse operation, the amplitude and segment widths are independently adjustable.

The Model 331-1 System enables the researcher to achieve the same measurement standards at substantially reduced cost in situations where a suitable corrosion cell system is already available. Except that it lacks the Model K0047 Corrosion Cell Kit, the Model 331-1 is the same as the Model 331-3 System described above.

The Model 331-2 System differs from the 331-1 System in that it incorporates a simple linear ramp generator, the Model 173/42, in lieu of the Model 175 Universal Programmer. Although less versatile than the Model 175 Programmer, the Model 173/42 is nevertheless suitable for many applications. The Model 173/42 provides scan rates over a range of 1 mV/minute to 5 V/minute.

All three systems include a Houston Model 2200-3-3 X-Y plotter for optimum display of the experimental results. Figure 2 illustrates how a system could be connected in a typical application.

SYSTEM COMPONENTS

- (1) MODEL 331-1
 - 1 Model 173 Potentiostat/Galvanostat with Model 178 Electrometer Probe
 - 1 Model 376 Logarithmic Current Converter
 - 1 Model 175 Universal Programmer with 175/99 Slow Sweep Option
 - 1 Houston Model 2200-3-3 X-Y Recorder All necessary cabling
- (2) MODEL 331-2
 - 1 Model 173 Potentiostat/Galvanostat with Model 178 Electrometer Probe
 - 1 Model 376 Logarithmic Current Converter
 - 1 Model 173/42 Voltage Programmer
 - 1 Houston Model 2200-3-3 X-Y Recorder All necessary cabling
- (3) MODEL 331-3
 - 1 Model 173 Potentiostat/Galvanostat with a Model 178 Electrometer Probe
 - 1 Model 376 Logarithmic Current Converter
 - 1 Model 175 Universal Programmer with 175/99 Slow Sweep Option
 - 1 Model K0047 Corrosion Cell System
 - 1 Houston Model 2200-3-3 X-Y Recorder

All necessary cabling

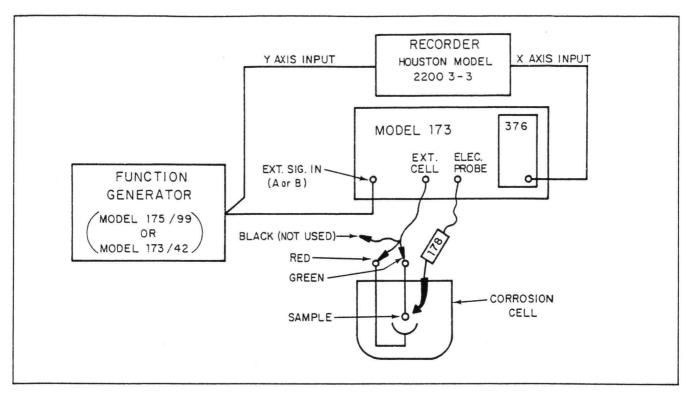


Figure 2. SYSTEM CONNECTIONS FOR TYPICAL CORROSION STUDY

CORROSION CELLS

Note that the system components provided with the Model 331-1 and with the Model 331-2 are flexible enough to be used with virtually any available corrosion cell system. Bear in mind, however, that the K0047 Corrosion Cell System provided with the Model 331-3 is ideally suited to corrosion investigations. Electrochemical measurements of corrosion phenomena require a cell system that is versatile, conveto use, and that can provide reproducible conditions from one experiment to another so that rational comparisons between specimens and/or environments can be drawn. The PARC Model K0047 Corrosion Cell System fulfills these requirements by incorporating the necessary cell, glassware, and hardware for performing rapid, accurate, and reproducible corrosion measurements. Unique features of this system are:

- (1) A 1 L flask with flat bottom to prevent tipping.
- (2) A leak-proof assembly for mounting specimens to be tested.
- (3) Twin high-density, non-permeable graphite counter electrodes.
- (4) A reference electrode bridge tube incorporating an ultra low leakage Vycor frit.

Figure 3 is a photograph of the Model K0047 showing the cell components and the cell connections for operation in conjunction with a Model 331 system.

CONNECTING THE SYSTEM

(1) Connect the output of the Function Generator to the

- EXT. SIG. IN connector of the Model 173 (there are two; either can be used). Be sure to set the toggle switch associated with the selected EXT. SIG. INPUT to the ON position.
- (2) The function generator output should also be applied to the recorder. In most applications, the function generator output is applied to the Y AXIS INPUT.
- (3) Connect the output of the Model 376 (should be plugged into the Model 173) to the recorder (X axis input in most cases). In applications where the current will vary over a wide range, use the Model 376 LOG I input. In applications that don't involve current varying over a wide range, the linear I OUTPUT can be used.
- (4) Connect the Model 178 Electrometer Probe cable and the Cell cable to the appropriate connectors at the front panel of the Model 173. NOTE: The power should be off and the Model 173 Cell Selector switch should be set to OFF when handling the cables and making the cell connections.
- (5) Assemble the cell. In the case of the PARC Model 9700, an instruction manual supplied with the cell describes its assembly in detail.
- (6) Make the cell connections. The red clip at the end of the cell cable should be affixed to the counter electrode (in the Model K0047 there are two counter electrodes that are connected together by a short lead). The green clip should be connected to the Working Electrode (sample). The black clip (ground) is

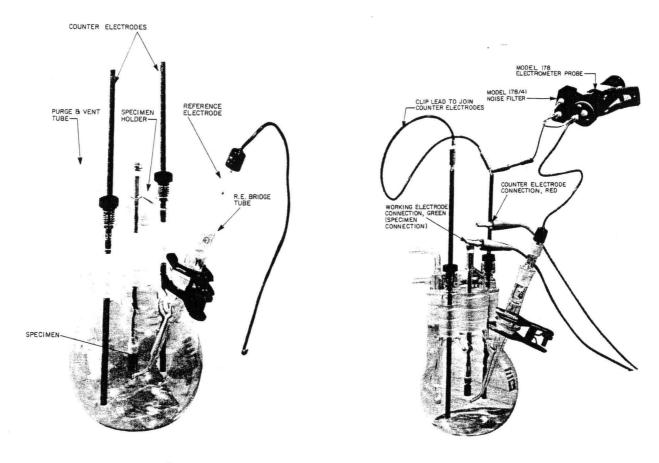


Figure 3. ASSEMBLED MODEL K0047 CORROSION CELL
AND CONNECTIONS THERETO

normally not used. If desired, it could be used to establish ground potential on a piece of apparatus. If it is not used, take care that it doesn't short against one of the active circuit elements.

- (7) Connect the Reference Electrode lead to the white pin jack on the face of the Model 178 Electrometer Probe.
- (8) If the Electrometer Probe is equipped with the Model 178/41 Noise Filter, connect a lead from one of the noise-filter pin jacks to the Counter Electrode. Connect the other noise-filter pin jack to the Reference Electrode. This connection is usually made at the white pin jack on the face of the Electrometer Probe.

This completes the assembly. Before operating, it is advisable to review the operating instructions in the Model 173 manual, in the Model 376 manual, in the Programmer manual, and, if appropriate, in the Model K0047 manual. Two points that should be kept in mind are: (1) the two Model 173 toggle switches associated with the Applied Potential/Current controls have no effect on the signal applied to the EXT. SIG. INPUT connectors; and (2) if using the Model K0047 Corrosion Cell System, bear in mind that the Vycor frit, once wetted, cannot be allowed to dry out. If it is allowed to dry more than a few minutes, it may crack and need to be replaced as described in the Model K0047 Instruction Manual.

NOTE: Users can obtain additional information on corrosion measurements by ordering the following application notes.

- (1) AN150: LOG CURRENT DENSITY MEASURE-MENTS WITH THE MODEL 331 CORROSION MEA-SUREMENT SYSTEMS
- (2) AN149: CALVANIC CORROSION MEASURE-MENTS WITH THE MODEL 173 POTENTIOSTAT
- (3) AN148: TAFEL PLOTS
- (4) AN140: LINEAR POLARIZATION
- (5) AN133: POTENTIODYNAMIC POLARIZATION MEASUREMENTS

APPENDIX C CALIBRATION AND USE OF X-Y RECORDERS FROM EG&G PARC

INTRODUCTION

Users of the Model 173 may have one of three variable-range X-Y Recorders, the Model 9002 Houston, the Model RE0074 Houston, of the Model 9012 Hewlett Packard. These instruments are virtually identical in their operating characteristics. The Houston is calibrated in inches and centimeters while the Hewlett Packard is calibrated only in inches. To short the input terminals, i.e., apply a potential of zero, the Houston calibration knob is switched to the position marked "ZERO". The same action is performed with the Hewlett Packard by pressing the button marked "CHECK".

Both recorders are pre-calibrated for several potential ranges. The ranges are labeled as MV/IN or V/IN. Both recorders have a 15 inch X-axis and a 10 inch Y-axis. Multiplication of the labeled potential ranges by these factors will give the full-scale potential range.

RECORDER RESPONSE TO APPLIED POTENTIAL

The M173 output is provided at a BNC connector. The center conductor of the connector carries the signal. The outer shell is usually grounded. The signal input to the recorder is made with a banana plug. One of the banana plug terminals is clearly labeled "GROUND" and is electrically attached to the outer shell of the BNC connector.

A "normal" instrument-recorder connection is made by inserting the "GROUND" banana clip terminal into the recorder terminal marked "-" (Houston) or LO (H/P). The other banana clip terminal is inserted into the recorder terminal marked "+" (Houston or HI (H/P). The recorder terminal labeled "GROUND" on the Houston is rarely, if ever, used in operation with the M173

With a "normal" instrument-recorder connection, a positive potential applied by the instrument to the recorder will cause the X-axis to move to the right and Y-axis to move in an upward direction. A negative potential will prompt the opposite action.

If the recorder terminal is reversed, i.e., "GROUND" to "+" or HI, a positive potential will cause the X-axis to move to the left and the Y-axis to move a downward direction.

RECORDER CALIBRATION WITH EG&G PARC INSTRUMENTATION

A recorder potential range other than those precalibrated may be desired for certain experiments, e.g., corrosion studies, cyclic voltammetry, coulometry, etc. The factors to be considered to calibrate and connect to the recorder correctly include the direction that the recorder will move for a given potential, the signal source for calibration, and the instrument that is used to drive the recorder during the experiment.

SIGNAL SOURCE

The Model 175 Programmer or the Model 173 Potentiostat can be used as a signal source for calibration. The recorder is attached to either the Model 175 "Signal Output" or the Model 173 summing amplifier output ("S/A OUT" on the rear panel). The desired signal can be obtained from the M175 by setting the "A" potentiometer and depressing "INITIAL".

The 173 operates somewhat differently, in that the potential at "S/A OUT" is of opposite polarity to that set on the front panel. It is not necessary to connect the electrode leads or switch to "EXT. CELL" to generate the signal. For example, put the M173 in "CONTR. E", set - 1.00 V on Channel A, and + 1.00 V will be output at "S/A OUT".

INSTRUMENT/RECORDER CONSIDERATIONS

The above comments also apply during the actual experiment. The potential axis of the recorder can be driven by the M173 Electrometer Monitor Output or the M175 Signal Output. The M175 Signal Output is that which is set on the M175. The M173 Electrometer Monitor Output provides a potential that is opposite in polarity to the potential that is being applied to the cell. Thus, one would calibrate the recorder differently for a M173 and a M175.

To take a simple example, assume one desired to scan from 0.00 to \pm 1.00 volts and display this on the recorder by starting at the far left and moving to the right along the X-axis as the scan progressed. A "normal" connection between the X-axis terminals and the M175 Signal Output would provide this type of scan. If the recorder was driven by the M173 Electrometer Monitor Output, the terminals would have to be reversed at the recorder, since the potential range of 0.00 to \pm 1.00 would be presented to the recorder.

It is essential, then, to be aware of the output characteristics of the signal to the recorder. This applies as well to the plug-in modules for the M173, where the signal for cell current is normally obtained.

RECORDER CALIBRATION

To calibrate the recorder at a range that is not preset, the first step is to set the recorder sensitivity at the next lowest setting. For example, to calibrate at 50 mV/in, set the sensitivity at 10 mV/in. The recorder will then be adjustable up to 100 mV/in. Then set zero at some point. When the proper signal is applied, adjust the recorder pen at the appropriate distance for the desired calibration with the potential adjust control. This is the inner knob on the "Cal/Var" switch on the Houston, and the inner knob on the sensitivity switch on the H/P. For example, by applying 0.500 volts and adjusting the pen to move 10 inches from the zero point, one can calibrate the recorder for 50 mV/in. The same calibration would be obtained by applying 0.250 volts and adjusting for a 5 inch pen deflection.

Once the recorder has been calibrated in the above manner, the calibration knob should not be touched. The pen can be moved to any point with the "ZERO" control without affecting the calibration.

INSTRUMENT-RECORDER INTERFACE FOR CURRENT MEASUREMENT

Generally, an electrochemical experiment involves the measurement of current as a function of potential. As mentioned previously, the potential axis of the recorder is driven by the M173 or the M175. The current axis is driven by the module that is plugged into the M173. These modules are the Model 176 I/E Converter, the Model 179 Digital Coulometer, and the Model 376 Log Current Converter. A voltage proportional to the measured cell current is available at a BNC connector on the front panel of each instrument. The Model 376 also provides a potential that is proportional to the logarithm of the cell current via a separate BNC connector.

The "I OUT" characteristics are the same for the M176, M179, and M376, with one exception. Each module has a 7-position switch for current ranges from 1 μ A to 1 A. Currents as high as 5 times that selected can be handled with no loss of accuracy by the M176 and M179. The overload capacity capability applies for all ranges except 1 A, when the maximum allowable current is 1 A. In the Model 376, the maximum allowable current is that set on the Current Sensitivity switch.

Experimentally, the current sensitivity should be one setting greater than the maximum expected cell current. For example, if the maximum cell current is 400 mA, the current range should be set at 1 A.

I or all modules, a current corresponding to full-scale (the setting on the Current Sensitivity switch) provides a voltage of 1 V at "I OUT". The polarity of the voltage at "I OUT" is negative for a reduction process (cathodic current) and positive for an oxidation process (anodic

current). The polarity of the meter on the M173, conversely, is negative for an anodic current and positive for a cathodic current.

For example, assume one wishes to measure the current during the reduction of Cu²⁺ to the metal. The Current Sensitivity setting depends on the amount of copper present. The Y-axis recorder sensitivity should be set at 0.1 V/in or 1 V full scale. The potential at "I OUT" will be negative since a cathodic current is being measured. Therefore, a "reversed" instrument recorder connection will provide a positive pen deflection for reduction currents. If we further assume that the Current Sensitivity on the module is set at 100 mA, then a one inch pen deflection will correspond to 10 mA.

The Model 376 Log Current Converter is capable of accurately measuring cell current over 5 orders of magnitude. A full-scale cell current corresponding to the Current Sensitivity setting will give 0 V at "LOG I OUT" on the front panel of the M376. The transfer function is +1 V/decade of current. A current of 10⁻¹ full scale will provide a voltage of +1 V at "LOG I OUT". A current of 10⁻⁵ full scale will give a voltage of +5 V at "LOG I OUT". The "LOG I OUT" potential is always positive, regardless of whether an anodic or cathodic current is being measured.

The "LOG I OUT" is primarily used in corrosion studies. The normal format is to present the current as increasing from left to right on the X-axis. To calibrate the recorder correctly, a "reversed" instrument-recorder connection is required. The pen is zeroed on the right side of the paper. The recorder sensitivity is set at 0.1 V/in. A potential of +5 V is applied and the pen is adjusted to the left-most vertical line with the potential adjust control. Alternatively, the recorder could be calibrated in exactly the same way with a "normal" connection and an applied potential of -5 V. When the recorder is attached to the M376 to begin the experiment, the recorder terminals must be changed to the "reversed" position. It should be noted that there is no difference between +0 V and -0 V.

APPENDIX D TRANSIENT SENSITIVITY

Generally speaking, the design and construction techniques used in equipment manufactured by EG&G PARC are conducive to assuring normal operation in the presence of moderate transient levels. Although these provisions are sufficient for operation in most places where this equipment is used, it is certainly possible for the 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, etc.
- (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.

If a user is having problems due to transient interference, or if operation in a high-transient environment is anticipated, steps should be taken to minimize the transient problem. Static discharge problems can sometimes be avoided by judiciously selecting one's clothing and shoes, or by altering 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 immediately prior to touching the instrument, particularly when making connections to it. Transients that enter the instrument via the line cord can generally be suppressed by means of external line-transient filters. Suitable devices are commercially available.