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BAS CV-37

February, 1984

MF-9031 INSTRUCTION MANUAL

Voltammograph

Bioanalytical
Systems, Inc

2701 Kent Avenue
West Lafayette
Indiana 47906

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FOR THE

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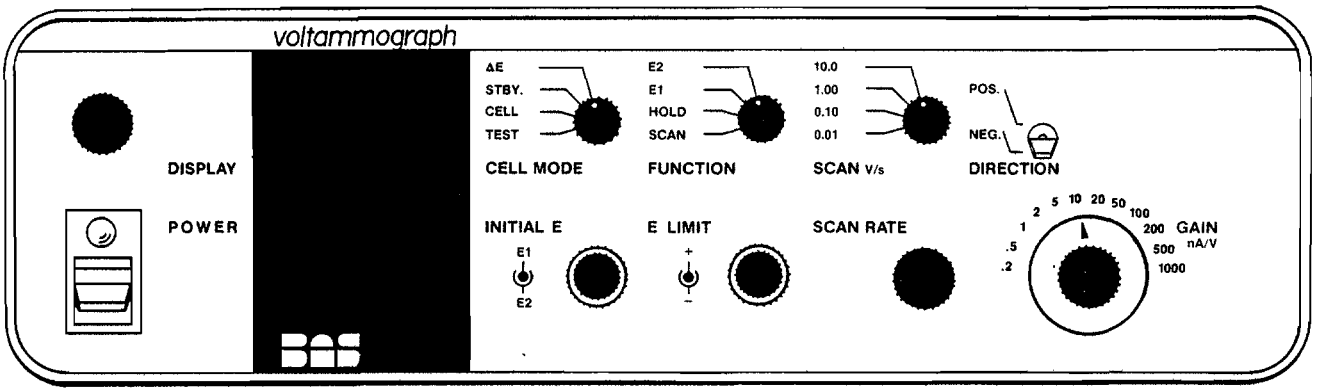
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OPERATION/INSTRUCTION MAINTENANCE MANUAL

FOR THE

CV-37
VOLTAMMOGRAPH



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BAS CV-37 Series P/N MF9030 02/24/84

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Section 1.0

The BAS model CV-37 Voltammograph is an electrochemical electronics unit capable of generating linear ramps and simple pulse potential waveforms across an electrode-solution interface and monitoring and amplifying the current resulting from subsequent electrochemical reduction-oxidation process. This instrument, when coupled with an appropriate electrochemical cell, can be used for a variety of electroanalytical techniques. Qualitative information of redox behavior by stationary electrode voltammetry (SEV) or cyclic voltammetry (CV) is easily accomplished using the CV-37 controller. Highly sensitive quantitative results can be obtained by using anodic or cathodic stripping voltammetry. A recent application of electrochemistry and one of the major reasons for developing this instrument is for in-vivo monitoring of neurochemically important compounds (see Section 2.1 for an overview of in-vivo electrochemistry).

This manual covers the installation and operation of the CV-37 Voltammograph, as well as, an introduction to voltammetry. It is designed to not only cover the physical manipulation of the knobs and switches to make the instrument work, but also introduce the user to some of the theoretical and practical background of the voltammetric experiment so that the data obtained can be intelligently interpreted. It is very important to have an understanding of the limitations as well as the advantages of the technique to form any conclusions from the resulting data.

1.1.1 Specifications and Features

Features:

Designed for neurochemical research, in-vivo monitoring of neurotransmitters.

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Compact.

Easy to use.

Waveform generator potentiostat, Current-to-voltage converter output signal processor all built into a single unit.

Can be externally controlled by timers or computers.

Front panel digital readout.

Specifications:

Applied Potential: E1 (Initial), E2 (Step)
Range: ± 5.00V
Compliance Voltage: ±10V
Maximum Current Available: 12 uA nominal
RC Time Constant: 0.01s
Linear Scan Rate Range: 0.1 mV/s to 10.0 V/s, continuously adjustable
Scan Direction: positive or negative going, manual selection
Scan Limit: positive and negative, continuously adjustable
Range: ±5.00V

Current to Voltage Output:

Range: 0.2 - 1000 nA/V, 1:2:5 sequence in 12 steps
Maximum Measurable Current: 12 uA

Semi-Derivative

$d^{\frac{1}{2}}i/dt^{\frac{1}{2}}$ ($I/t^{\frac{1}{2}}$ vs. E or t)
Range: Semi-derivative of ± full scale current output

Front Panel Controls:

Display Selector: Single knob
*Pos Scan Limit - ±5V
*Neg Scan Limit - ±5V
*Error-X Limits (Scan limits overlap) indicated on display
Scan Rate - 0.1 mV/s - 10 V/s and direction

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Temperature - 0-99.9 °C
Equilibrium (Rest) Potential (E)
- ±1.999V
Applied Potential (E1, E2) - ±5.00V
Current Out- 0.001 nA-1.99uA

Scan-Hold-E1-E2 (operation knob)
Test-Cell-Standby- E (operation knob, position indicated on display)
E1-E2 (one knob, dual concentric)
Scan Pos/Neg Limits (one knob, dual concentric)
Scan Range, (one knob, 4 position 0.01, 0.1, 1.0, 10.0 V/s)
Scan Rate, (one knob, variable adjust)
Scan Direction (toggle switch, +, -)

Current Range (one knob, 12 position 0.2-1000 nA/V)

Rear Panel Jacks and Switches:

Applied Potential, (Jack)
Polarity choice, (toggle switch,
+, -)
Current Output, (Jack)
Polarity choice, toggle switch, +
oxn., + rdn.)
Equilibrium (Rest) Potential, E (W-R_{grnd}), (Jack)
Auxiliary Input, Potentiostat, (Jack, Ext. In)
Semi-Differential Output, (Jack)
Filter (0.01, 0.1, 1 sec.), (toggle switch)
Temperature Input, (Jack)
Remote Control Connector (Used with external timing device)
E1 (On-Standby), E2 (On-Standby), Scan (On-Hold), Cell (On-Standby), Scan Direction (Pos - Neg),

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1.2 *Unpacking and Inspection of Equipment*

After carefully unpacking the instrument, check the contents against the inventory lists shown in the Tables in Section 1.2.1. Some items are shipped loose in the box e.g. manuals and small components packaged in plastic bags. To avoid losses, examine all packing material before discarding it.

If a shortage exists, call BAS Customer Service and describe the shortage. A replacement part will be sent immediately subject to stock availability.

If any damage has occurred whether obvious or concealed, all claims must be made to the carrier usually within 15 days of your receiving the shipment; otherwise the carrier may not honor the claim. Once the item has been assigned to a carrier for shipment, the carrier is responsible for its safe delivery. The following are some guidelines to follow when damage is discovered. You should contact the carrier directly for specific claim procedures.

United Parcel Service (UPS), Parcel Post, Air Parcel Post.

1. Retain container, packing material, and broken item for inspection by the carrier.
2. Write or phone BAS with our order number, the date received and a description of the damage. BAS will do everything possible to expedite repair or replacement of the items damaged.

Air Freight, Express or Truck

1. Contact the local agent of the transportation company immediately and request an inspection.
2. Retain the container, packing material, and damaged goods until the examining agent has made an inspection report.

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In all of the above cases, do not return damaged goods to Bioanalytical Systems without first contacting our customer service personnel for a Customer Service (Return Authorization) Number (CS#). When a defective part is returned to BAS, the CS number immediately identifies you as the sender and describes the item being returned. To avoid confusion, Bioanalytical Systems refuses all unauthorized return shipments.

1.2.1 Parts List

Part Number	Description
EF-1066	CV-37 Voltammograph
	Warranty Card
	Power Cord
	Recorder Leads (2 feet length)
	CV-37 Manual

Additional Accessories

MF-2103	Voltammograph Accessory Pkg.
MF-2020	RE-1B, Ag/AgCl Reference Electrode
MF-2055	RE-2, Calomel Reference Electrode

1.3 Warranty and Service Information

Bioanalytical Systems Inc. (BAS) products are fully warranted against defects in material and workmanship. Electronic instruments are unconditionally guaranteed for one year, except when failure is due to obvious abuse or neglect, unauthorized tampering, procedures not described in

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manuals, or improper connection of electronic units to other components. Electrochemical cells and electrodes are warranted for 60 days from date of invoice under the same exclusions.

Products sold but not manufactured by Bioanalytical Systems Inc. carry the original manufacturer's warranty. The following items are not covered under any warranty: carbon paste, activated aluminum oxide, lamps, panel lights, fuses, pump seals, or valve seals.

For any product expressly covered under this warranty, Bioanalytical Systems, Inc. is liable only to the extent of replacement of defective items. Bioanalytical Systems Inc. shall not be liable for any personal injury, property damage, or consequential damages of any kind whatsoever. The foregoing warranty is in lieu of all other warranties of merchantability and fitness for a particular purpose.

Service Information

Bioanalytical Systems provides a skilled service staff available to solve your technical problems if an equipment oriented problem should arise. For further details, call customer service personnel (317-463-4527) who will route your problem to the correct individual. Following discussion of your specific difficulties, an appropriate course of action will be described and the problem resolved accordingly. Do not return any products for service until a Customer Service Number (CS#) has been obtained. The CS# identifies you as the sender and describes the problem you are having in full detail. All correspondence and shipments should be sent to:

Service Department
Bioanalytical Systems Inc.
2701 Kent Avenue
West Lafayette, IN 47906

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Service will normally be completed within 10 working days. When calling for service and technical assistance, please have serial and model number information ready. This will speed the overall processing.

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Section 2. General Overview of In-Vivo Electrochemistry*

In the current practice of biomedical research it is frequently necessary to measure various substances in body fluids or tissue homogenates exterior to the living organism. While such information is extremely valuable in understanding the biochemical events associated with normally and abnormally functioning cells, organs, and complete organisms; it would clearly be highly desirable if these same types of measurements could be made in the intact biological system. With few exceptions the tools of modern analytical chemistry do not permit this. Chromatographs, mass spectrometers, and various optical instruments are clearly too large and complex to permit direct in vivo measurements.

Electrochemistry is one of the few methodologies available to us which is very amenable to miniaturization and implantation in living things. Unfortunately, while electrodes can readily be miniaturized to a reasonable degree, it is extremely difficult to bring them the selectivity necessary for reliable measurements in very complex systems. Furthermore, because electrochemical measurements inherently involve surfaces, surface active substances frequently "poison" the measurement. In a few cases (pH and O_2) it has been possible to bring adequate selectivity to small implanted electrodes. Even in these cases, serious difficulties remain for chronic implantation.

The understanding of the nervous system would clearly benefit tremendously from determination of transmitter substances and their metabolites in the intact organism. Correlation of behavioral and electrophysiological data with chemical information would be very exciting. It would be especially nice if such correlations could be done on a time scale comparable to the physiological events of interest. To date, this has not been possible. So-called push pull perfusion experiments have been useful, but remain experimentally exceedingly

* From "DCV-5 Voltammetry Controller" Operations Manual, 1979

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difficult to carry out and are often too slow.

About 10 years ago, Professor Ralph Adams at the University of Kansas began to explore the possibility of using miniature graphite electrodes to determine the catecholamines and their metabolites in vivo in unrestrained animals. Professor Adams undertook this challenging project because he was very familiar with the fact that phenolic compounds undergo facile oxidation reactions at carbon electrodes at physiological pH. At the time this work was begun, electrochemists typically employed electrodes of about 1-4 mm in diameter. Adams and his coworkers proceeded to prepare electrodes down to a diameter of about 100 μm (0.1 mm) and since that time even smaller electrodes have been prepared by his and other groups.

In 1972, the first successful use of such voltammetric electrodes in an unrestrained rat were carried out. As anticipated, there were serious difficulties with quantitating the results because of the poor selectivity of such electrodes. For example, it was immediately realized that such compounds as ascorbic acid, dopamine, norepinephrine, and DOPAC could apparently not be easily resolved using conventional electrochemical techniques. It was therefore judged in order to make useful measurements, one would have to place the electrodes in regions of the brain rich in a particular transmitter substance and/or carry out experiments which would more or less specifically influence the substances which could be measured with highest reliability. Various pharmacological and behavioral manipulations of animals were used to test this novel idea. Since that time, several other groups have gotten involved in the development of electrodes for implantation in the brain. Professors Lane, Wightman, and Justice are three young analytical chemists who have picked up the banner and are making some headway now. Serious technological difficulties remain. Besides the problem of selectivity, there are problems with reproducibility, with

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calibration, with poisoning the electrode surfaces, with tissue damage, and with control of the reference potential.

In spite of these difficulties, a number of rather interesting results have been obtained. At this point, however, it must be understood that electrochemical monitoring of transmitter substances in vivo is at a very early stage of development. Unfortunately, some active groups in neuropharmacology have perhaps gotten too excited about the technique, often without much appreciation of either the principles or the experimental practice of finite-current electrochemistry. Other groups have looked at the new technique with an extremely cynical eye. They have concluded that such measurements are impossible and that what has been done is largely of no value. It is certainly likely that no new neurological information has yet been obtained. Much of what has been done involves experiments to explore the validity of the technique! The potential importance of such measurements is so great that further development of in vivo electrochemistry is clearly warranted. This development needs to be done soberly.

Bioanalytical Systems serves the neurochemical community by manufacturing instrumentation for separation and detection of neurotransmitters and their metabolites based on the now well known LCEC technique. In order to stimulate further research in vivo electrochemistry, BAS has developed the CV-37 instrument. A very few of these instruments have been made. They have been sold to select laboratories at a price well below their cost as a service to the neurochemical community. BAS does not in any way imply that these instruments are capable of measuring individual compounds in vivo. We remain a neutral observer as the research unfolds. Lately there has been a great deal of controversy associated with (1) the type of electrodes to be used in these experiments and also (2) with regard to the electrochemical excitation function to be applied. For the former problem, various carbon paste, graphite epoxy, and

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carbon fiber electrodes are being promoted. In the latter are some workers using long term pulse techniques (chronoamperometry), some like fast chronoamperometry, others advocate linear scan voltammetry with or without semi-differentiation of the output, and still others insist that the experiment must be carried out with differential pulse voltammetry. The CV-37 is capable of chronoamperometry and linear sweep voltammetry with or without semi-differentiation. It is not capable of differential pulse experiments, which I personally believe to be unsuitable for in vivo measurements. In spite of my prejudice, the reason the instrument does not include this capability is that it would dramatically add to the cost and at this stage of development there are not many laboratories willing to invest a great amount of money to try in vivo electrochemical experiments.

At this stage, BAS will assist you in educating yourself about electrochemistry should you wish to make this investment. We will also put you in touch with other laboratories who are actively pursuing in vivo electrochemistry. At this time, we will not prepare electrodes for use with the instrument and as I mentioned, we will not vouch for the usefulness of the data obtained. We will guarantee, however, that the CV-37 is electronically sound and is capable of doing the electrochemical experiments for which it was designed. Electrochemistry is much more than electronics. The quality of the electrodes and the way they are used is often far more involved in the reliability of the data then is the electronics. We currently have no control over this.

The above editorial is an odd way to introduce a new product. I feel that it is essential for the user to understand why the CV-37 was developed and why it needs to be used cautiously. I am hopeful that my reservations will be removed as a result of quality research achieved with the instrument over the next few years. We at BAS will do everything we can to further research in vivo electrochemistry. It remains important that you recognize

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that these measurements are a subject of research in themselves and that it would be highly inappropriate to assume that in vivo electrochemistry is routine. As the field evolves, we will have a lot more information for you. Please keep us posted of your problems and your successes. I wish you the best in your pioneering work in this area.

Peter T. Kissinger, Ph.D.
President

Section 2.1 Potential Scan Techniques in a Stationary Solution*

Some of the most useful electroanalytical techniques are based on the concept of continuously varying the potential which is applied across the electrode-solution interface and measuring the resulting current. The present section describes large amplitude techniques in this category when the working electrode is immersed in a stationary solution.

Two voltammetric techniques, stationary electrode voltammetry (SEV) and cyclic voltammetry (CV), are among the most effective electroanalytical methods available for the mechanistic probing of redox systems. In part, the basis of their effectiveness is the capability for rapidly observing redox behavior over the entire potential range available. Since CV is an extension of SEV, many points pertinent to CV are discussed in the following SEV section.

Stationary-Electrode Voltammetry

In stationary-electrode voltammetry (SEV) the potential applied across the electrode-solution interface is scanned

*Some of this material is taken in modified form from the book Laboratory Techniques in Electroanalytical Chemistry, W.R. Heineman, P.T. Kissinger, Eds., Marcel Deker, NY, 1984.

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linearly from an initial value, E_i to a final value, E_f, at a constant rate, v (V/sec). This potential excitation is illustrated by the $E_{applied}$ time profile in Figure 2.1 for several rates of scan and several values of E_i and E_f. An excursion in which the potential becomes increasingly positive (i.e., is heading in a positive direction) is termed a positive scan (not an anodic scan). Since the time required for scan d is termed a negative scan since the potential is becoming increasingly negative (even though in this example the actual potential at any time is positive).

To obtain a stationary-electrode voltammogram, the current is measured during the potential scan in an unstirred solution. A voltammogram which was obtained with a Pt working electrode in a 1.00 mM Fe^{2+} solution with 1.0 M HCl supporting electrolyte is shown in Figure 1.2.

The potential scan which was applied to the working electrode to obtain this voltammogram is identical with profile b in Figure 2.1. The reader may correlate $E_{applied}$ in Figure 2.1 with the horizontal axis of Figure 1.2. Note that the experiment is completed in only one second.

In SEV one generally wants to initiate the potential scan at an E_i at which no electrolysis occurs and then scan through the standard electrode potentials of the species in solution. Thus, the composition of the reaction layer will not be altered in an unknown manner at the beginning of the experiment by application of E_i. The formal electrode potential of the Fe^{3+}/Fe^{2+} couple in 1 F HCl is ca. +0.46 V vs. SCE. Since the solution to be examined contained iron in the +2 oxidation state, an E_i which was negative of this potential was selected. This selection enabled the E_i to be applied across the electrode-solution interface without substantially altering the redox state of iron at the electrode surface. In order to do this, the E_i should be negative of the standard electrode potential by at least 177 mV for an n=1

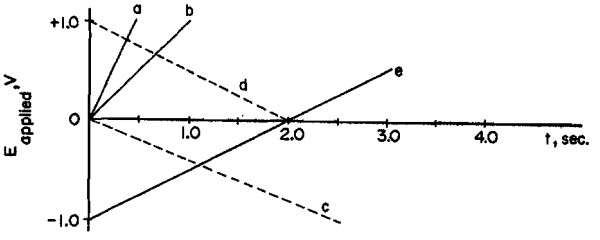


Figure 2.1 $E_{applied}$-time profiles for stationary electrode voltammetry.

Scan	E_i, V	E_f, V	ν, V/sec
a	0	+1.0	+2.0
b	0	+1.0	+1.0
c	0	-1.0	-0.4
d	+1.0	0	-0.5
e	-1.0	+0.5	+0.5

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reversible system. Note that selection of an E_i positive of the standard electrode potential, e.g., +1.0 V, would cause immediate conversion of Fe^{+2} to Fe^{3+} at the electrode surface (chronoamperometry) before the potential scan was initiated. Also note that in the case in which the iron is initially present in solution as Fe^{3+}, +1.0 V would then be a good selection for E_i.

In a practical vein, one does not always know what either the standard redox potential or the redox state is for the species in solution. In this case a potential can usually be found (by trial and error) which when applied does not cause current to flow. Such a potential is generally suitable for E_i since the absence of current (other than charging current) can be taken as evidence that no significant electrolytic perturbation of the reaction layer is occurring. One can then "find" the redox couple by scanning in either direction from this E_i.

In our Figure 1.2 example, after imposing E_i across the electrode-solution interface, the potential is scanned positively toward the standard redox potential of the Fe^{3+}/Fe^{2+} couple. The ratio of iron redox states which must exist at the electrode surface at several potentials during the scan are shown on the potential axis. These values are dictated by the Nernst equation for a reversible system. Since the solution initially contained "only" Fe^{2+}, the Fe^{3+} required to satisfy the Nernst equation is obtained from Fe^{2+} by oxidation, causing current to flow.

The physical situation in the solution adjacent to the electrode during the potential scan is illustrated by the concentration-distance (C-d) profiles included in Figure 1.2 for selected times. Profile a is the C-d profile of Fe^{2+} when E_i is imposed. Note that the application of E_i did not measurably alter the concentration of Fe^{2+} at the electrode surface as compared to the solution bulk. As the potential is scanned positively, the concentration of Fe^{2+} at the electrode surface decreases in order to establish a

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Fe^{2+}/Fe^{3+} ratio which satisfies the Nernst equation for the applied potential at any particular instant. This is illustrated by profiles b-e. Note that profile c (for which the concentration of Fe^{2+} at the electrode surface exactly equals the concentration of Fe^{3+}) corresponds to an $E_{applied}$ which equals the formal electrode potential (vs. SCE) of the couple.

Profiles e-g correspond to potentials sufficiently positive of the formal electrode potential that the concentration of Fe^{2+} is effectively zero at the electrode surface. The conditions for these profiles are identical with those for chronoamperometry. Once the potential has reached a value sufficient for a zero reactant-surface-concentration, the potential and its rate of change become immaterial to the diffusion controlled current. In other words, should the scan be stopped at f, the current will follow the same time course as if the scan had been continued.

The behavior of the current during the potential scan can be understood by carefully examining the C-d profiles in Figure 2.2. The current is determined by the slope of the C-d profile for the electroactive species at the electrode surface, one can correlate the magnitude of the slopes of the profiles in Figure 2.2 with current on the voltammogram at those potentials. The slope of profile a is zero and no current flows at that potential. As the potential is then scanned positively, $(\partial c/\partial x)_{x=0}$ increases for profiles b-e and the current in Figure 1.2 increases correspondingly. However, when profile e is reached, i.e., chronoamperometric condition of diffusion controlled conversion of Fe^{2+} is achieved, $(\partial c/\partial x)_{x=0}$ decreases as shown by profiles g and h because of depletion of Fe^{2+} near the electrode. Correspondingly, the current now drops with a $t^{-\frac{1}{2}}$ dependence as in chronoamperometry. Thus, the observed current behavior for the voltammogram is an increase to a peak current which corresponds to achievement of diffusion control at which point the current decays.

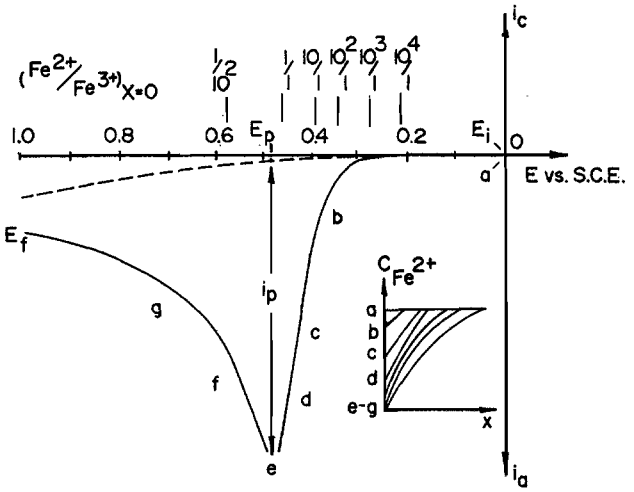


Figure 2.2 Stationary-electrode voltammogram for 1 mM Fe^{2+} in 1 F HCl at a Pt electrode. Electrode reaction is $Fe^{2+} \rightleftharpoons Fe^{3+} + e$. (----) SEV for 1 F HCl without Fe^{2+}.

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Two important parameters in SEV are the peak potential, E_p and the peak current i_p, which are the potential and current at the characteristic peak as shown in Figure 2.2. For a reversible system the peak current is defined by the Randles-Sevcik equation:

$$i_p = 269n^{3/2}AD^{1/2}C_b v^{1/2} \qquad (2.1)$$

at 25°C where i_p is the peak current, amperes; n is the number of electrons transferred; A is the electrode area, cm^2; D is the diffusion coefficient of the species being oxidized/reduced, cm^2/sec; C_b is the concentration of this same species in the bulk solution, mol/l; and v is the scan rate in V/sec. The peak potential is defined (for a reduction) by

$$E_p = E^\circ_{O,R} - \frac{0.029}{n} \qquad (2.2)$$

at 25°C where E_p and $E^\circ_{O,R}$ are expressed in volts, where D, E_p, and $E^\circ_{O,R}$ is the standard electrode potential corrected to the reference electrode being used.

According to equation (2.1) i_p is proportional to $v^{1/2}$ and a plot of ip vs. $v^{1/2}$ should be a straight line for a reversible system. Although the peak current increased with scan rate, the potential at which the peak occurs is invariant with scan rate as indicated by equation 2.2. The reason for the increase in current with faster scan rate is illustrated in Figure 2.3.

Figure 2.3 shows voltammograms and the C-d profiles for SEV at two scan rates. Comparison of the profiles for the faster scan rate b with the profiles for a scan a reveal a difference in the steepness of the profiles at x=0. For any given potential $(\partial c/\partial x)_{x=0}$ is larger for the faster scan. Consequently, the current is larger. In both cases, the value of the concentration at the electrode surface (x=0) is determined by the Nernst equation and is therefore independent of the scan rate. However, in the faster scan, less time is available for consequent depletion of the adjacent solution, resulting in

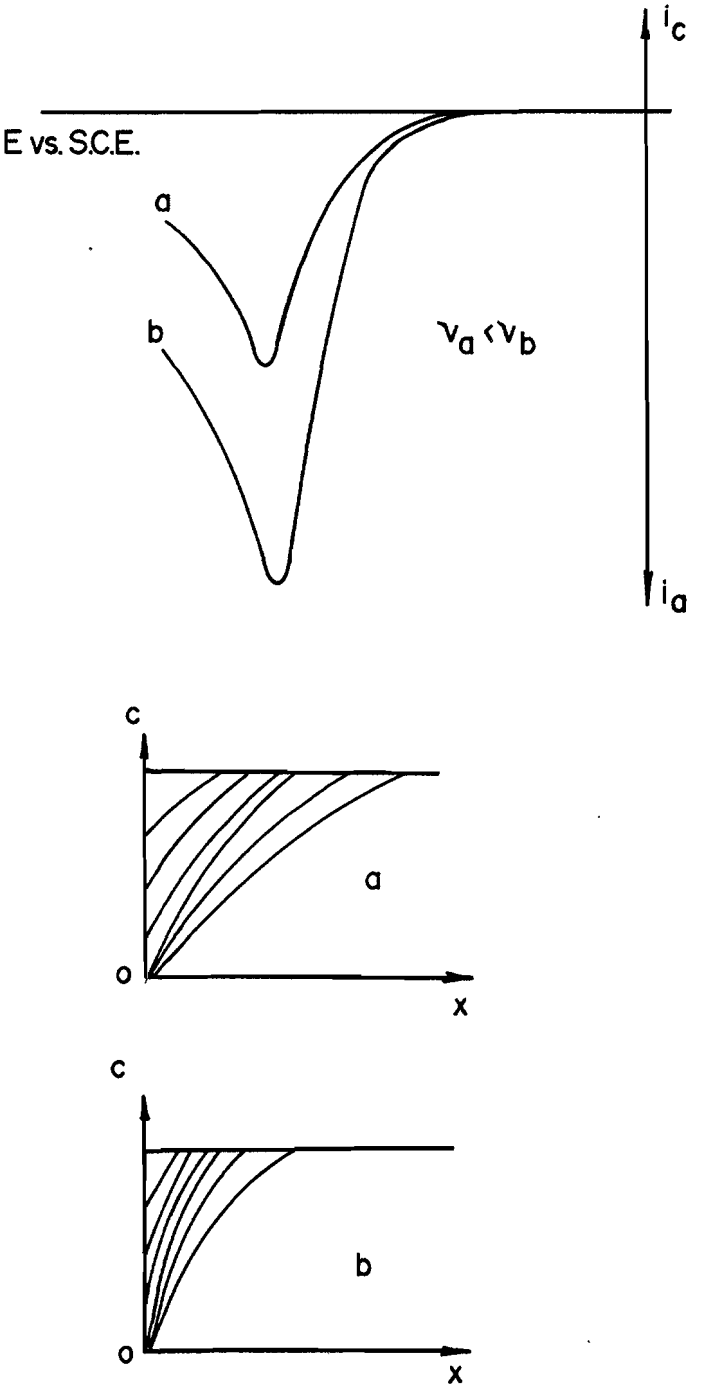


Figure 2.3 Voltammograms and C-d profiles for two different scan rates.

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steeper profiles. [If the current for both scans is integrated where

$$\int_0^t i\,dt = Q$$

for which scan rate will Q (the total charge passed) be larger?]

In the case of an irreversible system, the equation for i_p is

$$i_p = 3.01 \times 10 n(\alpha n_a)^{1/2} A D^{1/2} C b_v^{1/2} \qquad (2.3)$$

where α is the transfer coefficient and n_a is the number of electrons in the rate determining step of the electrode process. The peak potential, E_p, is no longer independent of scan rate as shown by equation 2.4.

$$E_p = E^\circ + \frac{RT}{\alpha nF}\left[-0.78 + \ln\frac{K_l}{D_O^{1/2}} - (1/2)\ln\left(\frac{nF}{RT}v\right)\right] \qquad (2.4)$$

where k_l is related to the heterogeneous rate constant of the electron transfer reaction. In general, irreversible behavior gives voltammograms which are more spread out. Figure 2.4 shows the influence of an n_a on the appearance of voltammograms.

An important aspect of voltammetry is the background current which is the current obtained on a solution containing all ingredients except the electroactive species. This current is designated by the dotted line in Figure 2.2. It is composed of (1) residual current, oxidation or reduction of adventitious electroactive material (e.g. dissolved oxygen, the solvent, or the electrode itself) and (2) charging current, non-Faradaic current required to charge the electrode to a given potential (behavior analogous to a capacitor). The peak current should be measured in a manner which does not include the background current. A common technique is to extrapolate the baseline current which precedes the Faradaic current and measure i_p by difference. Alternatively, a separate scan (at the same scan rate) can be made on the supporting electrolyte solution. Here i_p is the difference between the peak current (measured from

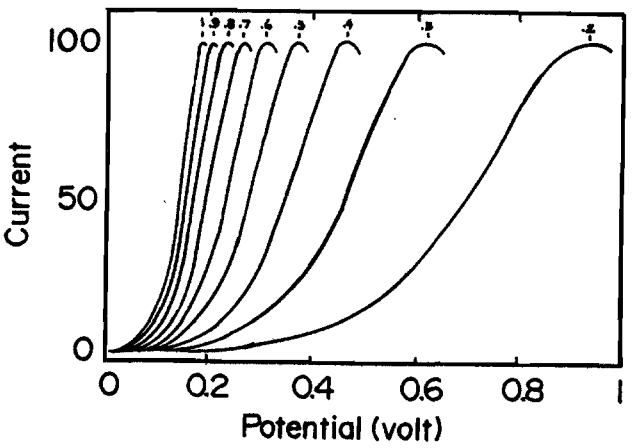


Figure 2.4 Voltammograms for various values of αn_a.

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zero current) and the background current at the same potential as shown in Figure 2.2.

The current due to charging the electrode to a given potential is directly proportional to the scan rate as given by equation (2.5)

$$i_c = C\frac{dE}{dt} = AC_{d.l.}v \qquad (2.5)$$

where i_c is the charging current, A is the electrode area in cm^2, and $C_{d.l.}$ is the capacitance of the double layer per cm^2. Consequently, the correction for this component of the background current becomes increasingly significant as the scan rate increases. It is important to recognize that $C_{d.l.}$ is a function of potential and therefore i_c is a function of E as well as v.

As a technique for analysis, SEV is reasonably sensitive, being useful at 10^{-5} M in some cases. Sensitivity can be improved by increasing the scan rate which increases the magnitude of i_p as in equation (2.1). However, charging current becomes a problem at higher scan rates since i_c increases more rapidly with v than does i_p ($i_c \propto v$, $i_p \propto v^{1/2}$). This significant charging current can be compensated for by a differential technique in which simultaneous scans are performed on two matched electrodes, one immersed in sample and one in supporting electrolyte. The background current from the supporting electrolyte scan is electronically removed from the voltammogram of the sample. The sensitivity of the rapid scan and differential SEV techniques is better than that of conventional polarography but not as good as that of the current sampled pulse polarographies.

The sensitivity of SEV for analysis is greatly extended by its use in conjunction with an electrochemical preconcentration step. This technique is termed DC stripping voltammetry.

SEV is a very effective means of probing homogeneous chemical reactions which are coupled to electrode reactions, especially when it is extended to cyclic voltammetry. Considerable information can

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be obtained from the dependence of i_p and E_p on the rate of potential scan. Figure 2.5 illustrates the behavior of i_p and E_p with variation in scan rate for a reversible heterogeneous electron transfer reaction which is coupled to various types of homogeneous chemical reactions. The current function ψ_p is proportional to i_p according to the equation

$$\psi_p = \frac{i_p}{nFAD^{1/2}C\left(\frac{nF\nu}{RT}\right)^{1/2}} \qquad (2.6)$$

Different types of coupled chemical reactions (or mechanisms) can be identified by their characteristic current and potential behavior with varying scan rate as shown in Figure 2.5. Theory and diagnostic criteria have been developed for a variety of kinetic cases in which the homogeneous chemical reactions are coupled to either reversible or irreversible heterogeneous electron exchange. Adsorption of the electroactive species has also been considered.

In general, conclusions based on current measurements are more reliable than those based on potential measurements because experimental factors (iR drop, function potentials, state of electrode surface) can shift the potential axis significantly.

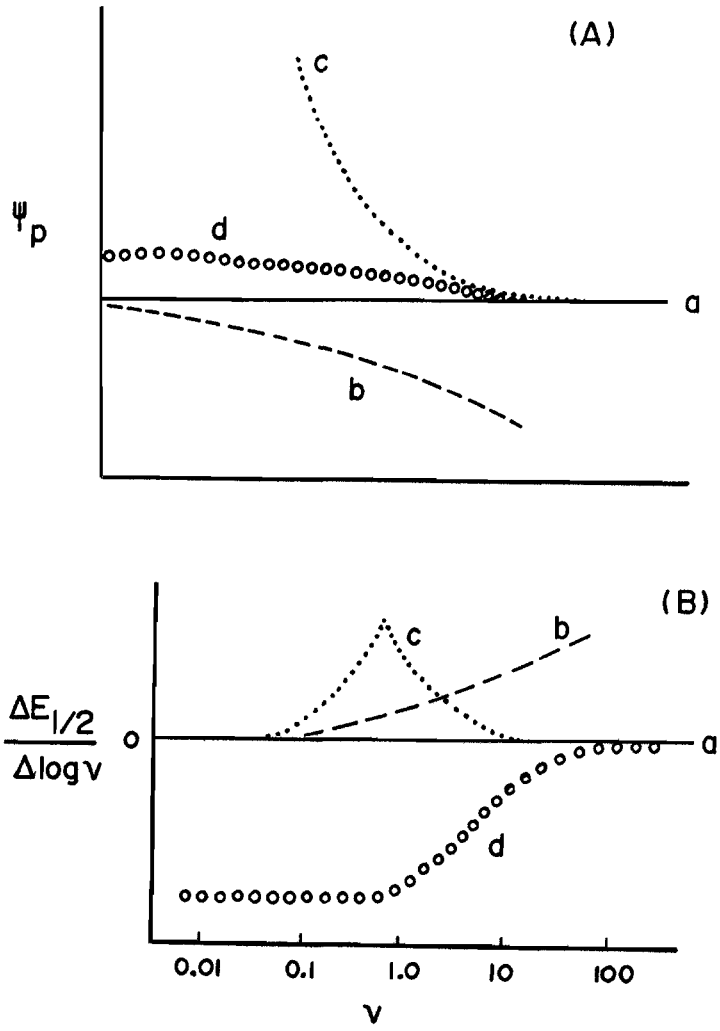


Figure 2.5 The influence of ν on (A) i_p and (B) E_p for several mechanisms.

(a) $O + e^- \rightleftharpoons R$, rev.

(b) $Z \rightleftharpoons O$
$O + e \rightleftharpoons R$, rev.

(c) $O + e^- \rightleftharpoons R$, rev.
$R + Z \longrightarrow O$

(d) $O + e^- \rightleftharpoons R$, rev.
$R \longrightarrow Z$

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Section 3.0 Installation

This section details the physical requirements for installation of the CV-37.

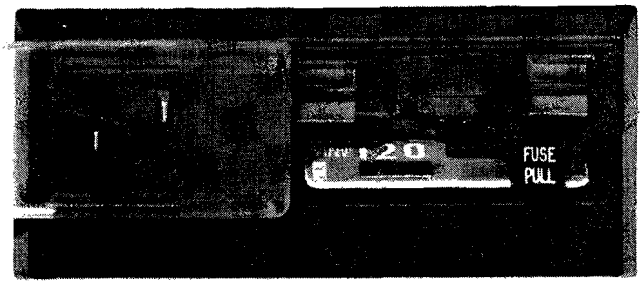
3.1 Power Requirements and Connection of the Power Cord

The CV-37 can be powered by any of the following AC voltages:

100 V
120 V
220 V
240 V

and frequencies of either 50 or 60 Hz. The power supply in your location (i.e. any of the above volts AC combinations) must be the same as the voltage selected in the power cord connector on the rear panel. The CV-37 is shipped with the voltage option already matched to the power requirements of the destination.

Caution:
To Prevent Electrical Shock. DO NOT Remove Cover
No User Serviceable Parts Inside Refer Servicing
To Factory Personnel.



Should the power option need to be changed, unplug the line cord and slide the plastic window of the power cord inlet to the left. See Figure 3.1. The orientation of the small circuit board now exposed in the socket determines the voltage option. If the voltage labeled on the outer edge of this board is not that required, pull out the card, and turn it (either by rotation or inversion) such that the desired voltage is readable. Reinsert the board and push the fuse holder back into the cavity. As illustrated in Figure 3.1, the board is oriented for 120 V, 60 Hz operation.

If the instrument is operated from a power outlet without safety ground connection, an appropriate adaptor should be used. The ground connection of this adaptor must be securely fastened to an external earth ground for safety purposes. The maintenance of a proper ground connection is also very important in the performance of this instrument. A proper ground connection cannot be overemphasized.

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The actual power cord is shipped as a separate item and must be plugged into the connector on the rear panel. To insert the power cord into the power socket, slide the plastic window to the right (i.e. over the voltage selection circuit board) and plug the female end of the power cord into this connector. The male end of the power cord is plugged into the power outlet socket. Note, the plug type on the male end of the power cord may vary depending on the country of destination.

3.2 Cell Connections

The cell lead is the group of wires that connect the CV-37 electronics to the electrodes of the electrochemical cell. One end of this lead plugs into the rear panel of the CV-37 and the other end terminates with the appropriate connectors for attaching directly to the electrodes of the cell.

3.2.1 CV-37 To Cell Cable

The proper connection of the cell lead is shown in Figure 3.2. The stainless steel connector on the cell lead is inserted into the plug mounted on the rear panel of the unit. Insert the cable connector into the mounted plug and rotate until the cable connector easily slides into the mounted connector. Continue pushing until the pieces "snap" together. To remove, simply pull on the cable connector. The catch will easily and automatically release.

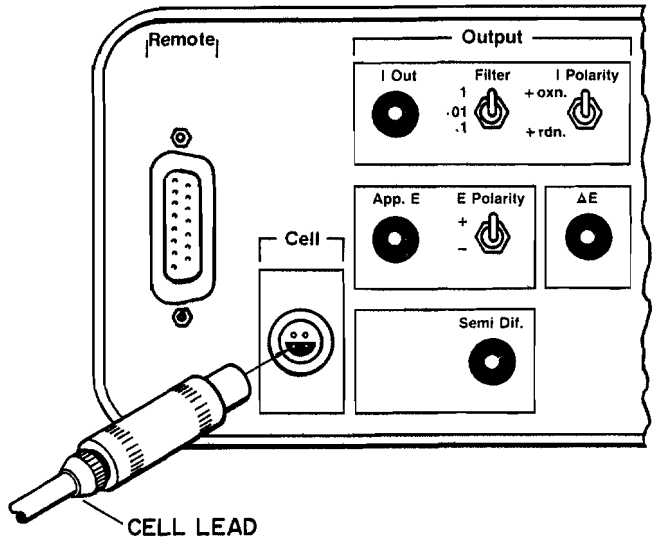


Figure 3.2
Connection of CV-37 to the Cell Cable

3.2.2 Cell Cable to Cell (Electrodes)

A variety of electrochemical cells with variable designs are available commercially. A very versatile electrochemical cell accessories package includes a mounting stand (with stirring and gas purging capabilities), vials, electrodes, etc. is the BAS Voltammograph Accessories Package, P/N MF-2013. In the package the cell stand, BAS model C-1B

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contains the cell cable that attaches directly to the CV-37 as described in the previous section. Connection of the cable wires to the individual electrodes is described in the cell stand manual. Note that the CV-37 contains a general purpose cell cable that terminates with connectors that can directly be attached to cell electrodes. The cable contains four color coded connectors (wires), three electrode leads and one grounding (shielding) lead with alligator clips on the ends. The color code is:

Black covered wire: working electrode lead.
Red covered wire : auxiliary electrode lead
White covered wire: reference electrode lead
Bare wire or Black wire with solder lug: ground connector

A strain relief connector i.e. a plastic mounting lug near the end of the cell cable, provides a point to affix the cable to prevent jostling of the cell when not in use. Figure 3.3 illustrates the cell end of the CV-37 cell cable.

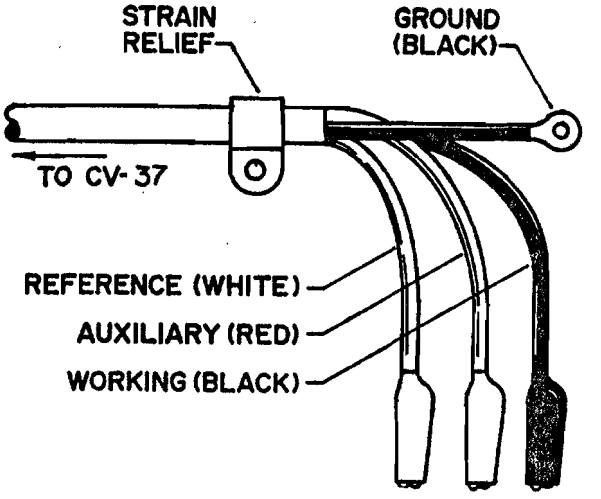


Figure 3.3
Cell (Electrode) End of Cell Cable

3.3 Recorder Connections

Two sets of recorder leads are provided with the CV-37 unit. One set is a single two feet length cable with a green and black banana plug on one end and Y-shaped lug on the other. Usually the banana plugs are connected into the CV-37 and the lugs are attached to the recorder.

The recording device must be capable of handling the input of analog voltage signals; typically 0 to ± 1 V.

The recorded output is usually a response function versus excitation function or time. The response function from the CV-37 could be current, potential, or the semi-differential of the current vs either time or the excitation function, usually applied potential.

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These functions are typically displayed on an X-Y or Y-T (time base) recorder, the BAS models RXY, P/N MF8010 and the model RYT P/N MF8000, respectively. A typical Y-T recorder connection is shown in Figure 3.4. The black banana plug (i.e. black coated wire) is plugged into one of the **Common** jacks on the rear panel of the CV-37. The green plug is plugged into the appropriate or required output response you want to record.

In the case where a response is plotted versus a specific excitation, a X-Y recorder will be required and the proper connection in such a case is shown in Figure 3.5. Generally the response is plotted on the Y-axis and the excitation on the X-axis. One of the cables provided is used to plot the Y response and the other is used to plot the X excitation. The green plugs are plugged into the appropriate CV-37 jacks on the rear panel and the Y-shaped lugs are attached to the posts or jacks on the recorder. Note, the black wire on either cable is always the commmon (or low) output from the CV-37 to the low input (sometimes labelled as "-" or ground) on the recorder. This input to the recorder has a jumper wire attached to it which is used to connect to an "earth ground" or a house common which connects to your recorder via the ground pin of the three pronged wall (power) receptacle or source. This jumper on these cables must be connected for proper operation of the CV-37. This is illustrated in Figure 3.5.

On the rear panel of the CV-37, an output polarity option is available. As is noted, output current responses are either positive for oxidations (+ **OXN**), i.e. an oxidation current causes a positive voltage output signal, cathodic currents are negative) or positive for reductions, + **RDN** (i.e. reduction current causes a positive voltage response, oxidation currents are negative). The latter case is the more conventional sign designation. The polarity out of the applied potential (**E Polarity**) can also be chosen. The "+" option yields an output which is most positive to the right on the

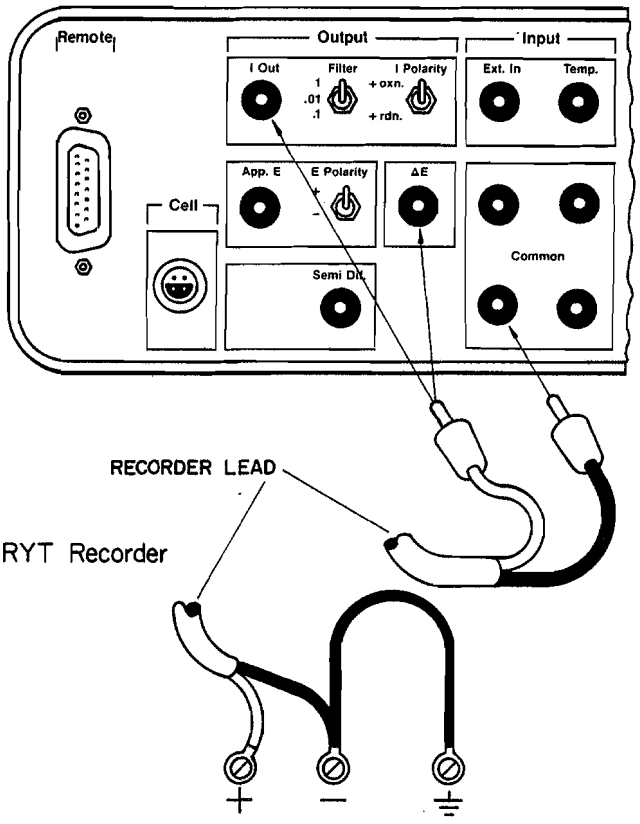


Figure 3.4
Connection of the CV-37 to Y-T Recorder

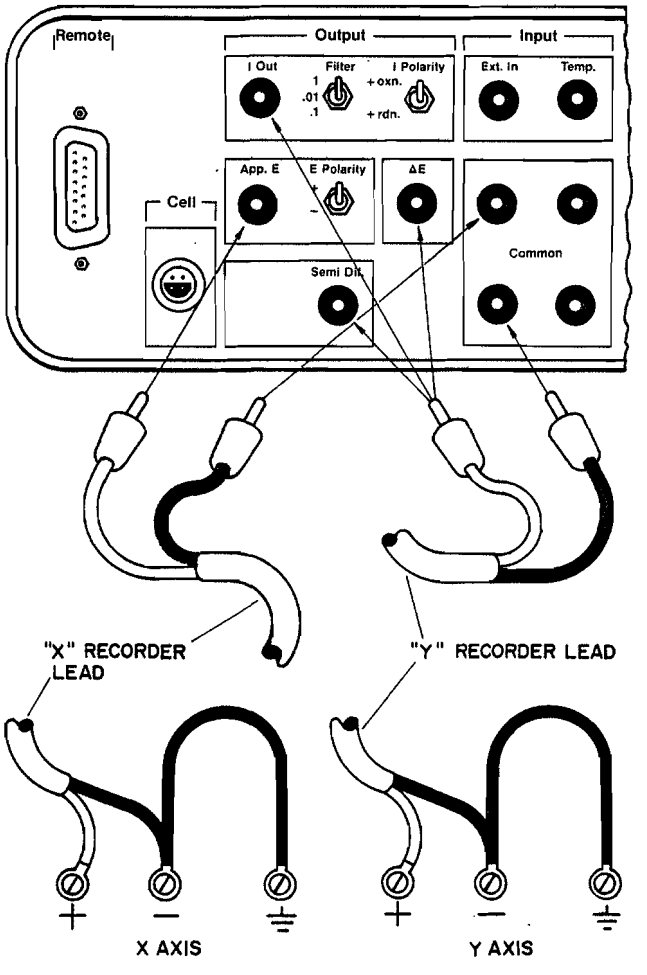


Figure 3.5
Connection of the CV-37 to X-Y Recorder

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X-axis (or up if the applied potential is used as the Y-axis). This output is inverted when the "-" option is chosen.

3.4 Auxiliary (External) Input

This input allows an external voltage source to be summed into the voltage of the internal potentiostat. For example, a waveform generator may be used to bring in a sine wave at a particular frequency for a specific application. The limits on this are the same as for the potentiostat as outlined in Section 1.1.1.

This input simply requires a cable with a banana plug on one end and the other end attached to the voltage generator. Of course, the circuit is completed by plugging into one of the **Common** jacks.

3.5 Temperature Measurement Input

The CV-37 in combination with the BAS model LC-22A Temperature Controller, auxiliary temperature probe and connecting cable, P/N EF1040, EW8116, EW8107 respectively, can be used to display and moitor the temperature from, for example, the electrochemical cell. The proper connection of these units is shown in Figure 3.6.

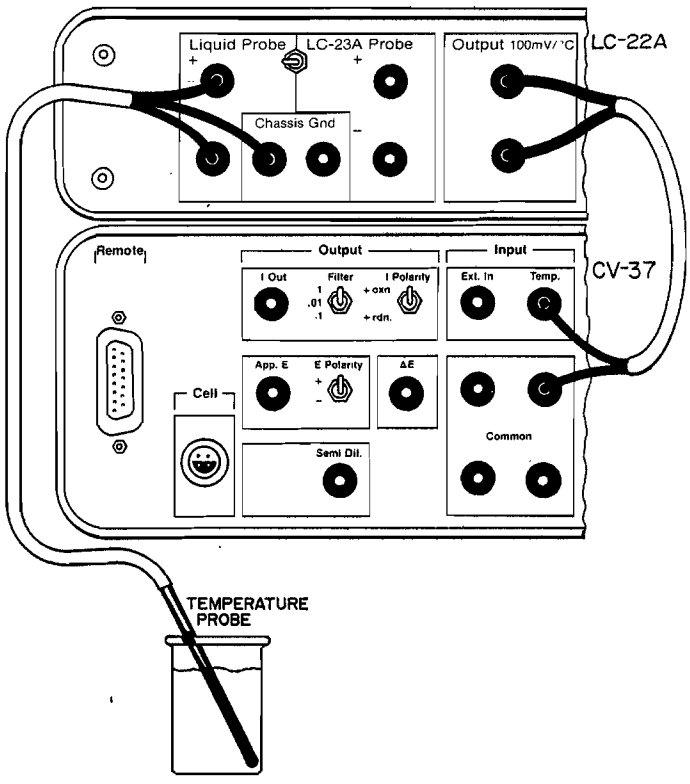


Figure 3.6
Connection of the CV-37 to the LC-22A Temperature Controller and Liquid Temperature Probe

The auxiliary temperature probe i.e. Liquid Probe, plugs directly into the color coded and labeled jacks on the rear panel of the LC-22A Temperature Controller. The connecting cable plugs into the output, labeled **Output, 100 mV/°C**, of the Temperature Controller and the input labeled **Temp Input** on the rear panel of the CV-37. The CV-37 is appropriately calibrated so that the actual temperature at the probe is read directly on the front panel display.

Note the LC-22A is a temperature controller and as such can provide power to heat up auxiliary heater units such as

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a knift heater, heating mantle, etc. and will control that temperature to ±0.1°C.

3.6 Remote Timing Input

The following Operational Modes can be controlled remotely:

Cell (on/off)
Scan (on/hold)
E1 (on/standby)
E2 (on/standby)
Scan Direction (pos/neg)

Any contact closure to ground will activate these functions.

The connector, BAS model EF1067, is attached to the rear panel as shown in Figure 3.7 and the pin designations are as follows:

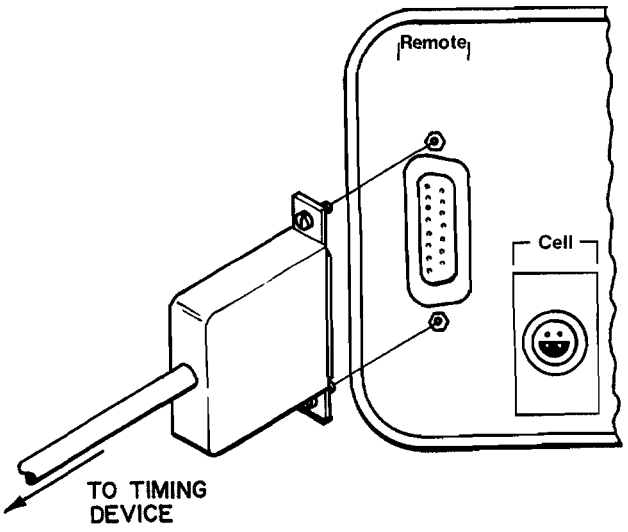


Figure 3.7
Connection of Remote Control Input

Operational Function	Low/ High	Pin	Color Code
Digital ground		1	Black
Scan	on/hold	2	Brown
Cell	on/stby	3	Red
E2	on/hold	4	Orange
E1	on/hold	5	Yellow
+ DIR	on/off	6	Green
- DIR	on/off	7	Blue

The timer can be any that meet the above conditions. There are a number of such units on the commercial market.

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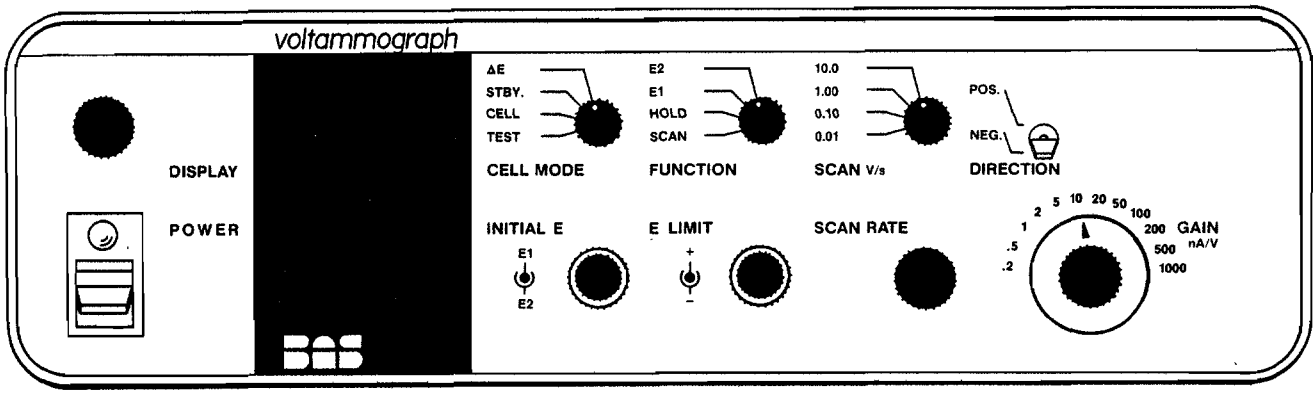
Section 4.0 Operation

This section describes and discusses the actual operation of the controls and outputs of the CV-37.

4.1 Identification of Controls/Outputs

4.1.1 Front Panel

The front panel switches and display are used for both control and operation functions of the CV-37. The front panel is shown in Figure 4.1.



4.1.1.1 POWER

This switch applies main electrical power to the instrument circuitry. When this switch is pushed up, a small red lamp directly above the switch will become illuminated indicating that power is being supplied. When turning on (or off) the main power, make certain the **CELL MODE** switch, which is discussed in Section 4.1.11, is in the standby, **STBY**, position.

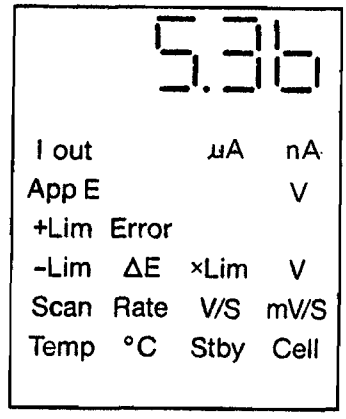


Figure 4.1
Front Panel of CV-37

4.1.1.2 DISPLAY

The **DISPLAY** knob is used to chose the function, whether control or output, displayed on the meter. There are eight positions on this knob thus eight functions that can be displayed. By rotating this knob the various functions and their corresponding numerical values

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and units are observed in the display window. There are 17 indicators (function, status, functional units) in the display log. If all indicators were lighted at the same time, determining which function is actually being displayed would be very confusing. To avoid this possible confusion when looking at the display window, the display brightly illuminates the function (control or output) that is being monitored on the meter, and on switching the display knob to a new position, the new function is brightly lighted but the other seven functions are dimly lighted, and fade out to dark after approximately 5 seconds. Thus, the operator knows how many switch positions the knob must be advanced to get to the desired function.

The display functions are as follows:

I Out is the output current in microamperes, **µA** or nanoamperes, **nA.**

App E is the applied potential in volts, **V.** This potential reading could be control information or output information depending on the **FUNCTION** chosen. The **FUNCTION** control will be described in a following section.

+ Lim is the most positive (this could be a negative number) potential (i.e. positive limit) in volts, **V,** in which the potential scan is reversed or switches direction from positive going to negative going.

-Lim is the most negative (this could be a positive number) potential in volts, **V,** in which the potential scan is reversed or switches direction from negative going to positive going.

If the **+ Lim** potential is adjusted such that it is less than (i.e. more negative) the **- Lim** value or the **- Lim** is adjusted more positive than the **+ Lim,** two display indicators are illuminated, the **Error** and **XLim.** As these indicators state, this is an error since the potential scan limits, **Lim,** are crossed,

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X. The corrective measure is to either adjust the **- Lim** or the **+ Lim** until the lamps shut off.

Scan Rate is the change of the applied potential with time in units of volts/seconds **V/S** or millivolts/second, **mV/S.**

Temp is the temperature in **°C** as monitored through the LC-22A Temperature Controller and the "Liquid Probe" transducer.

4.1.1.3 INITIAL E

The **INITIAL E** is a dual function knob used to vary or adjust two potentials, **E1** and/or **E2.** **E1** is adjusted with the outside knob and **E2** is adjusted with the inside knob. To set **E1**, the **FUNCTION** switch is at **E1** and the **DISPLAY** is monitoring **App E.** Simply turn the outside knob until the desired value between -5.00 V and +5.00 V is shown in the display window. **E2** is set in the same manner except the **FUNCTION** switch is selecting **E2** and the inside knob is turned to adjust to the desired potential.

4.1.1.4 E LIMIT

The **E LIMIT** is a dual function knob used to adjust the most positive and the most negative potential scan limits or the potentials where the scan direction reverses. This function is primarily used in the technique of cyclic voltammetry. The most positive limit i.e. **+ Limit** is set by selecting the **+ Lim** function in the display window and turning the outside, **+**, knob. This value can be between -5.00 V and +5.00 V but not less than the negative limit, **- Lim**, potential. If the limits cross and an error signal (**Error, XLim**) is shown in the display window, then either the positive limit, **+ Lim** must be made more positive or the negative limit, **-Lim** must be made more negative. The negative

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limit is adjusted in the same way as the positive limit except the **- Lim** is selected as the display function and the inside knob is turned to adjust to the required value between -5.00 V and +5.00 V. As with the positive limit adjustment, if the negative limit crosses the positive limit, the error signal, **Error, XLim**, will be shown in the display window and one of the two limits must be changed to remove the **Error** status.

4.1.1.5 SCAN V/s

The **SCAN V/s** switch is used to choose the relative order of magnitude of the rate of change of potential i.e. the potential scan rate. One of the four positions can be chosen. The **0.01** position for scan rates from 0.1 to 10.0 mV/s, the **0.10** position for scan rates from 10.0 to 100.0 mV/s, the **1.00** position for scan rates from 0.10 to 1.00 V/s, and the **10.0** position for scan rates from 1.0 to 10.0 V/s. The actual scan rate value required that is between these noted limits is controlled by the **SCAN RATE** switch discussed in the following section, 4.1.1.6.

4.1.1.6 SCAN RATE

The **SCAN RATE** knob is a variable adjust control for the rate of change of potential i.e. scan rate. To adjust to the desired scan rate, the **Scan Rate** function is selected in the display window by the **DISPLAY** knob, the correct relative magnitude of the scan rate is chosen by the **SCAN V/s** control switch as described above in Section 4.1.1.5, and finally the **SCAN RATE** knob is turned until the desired value is observed in the display window.

4.1.1.7 DIRECTION

This momentary contact toggle switch determines the potential scan **DIRECTION**, either positive going direction, **+**, or negative going direction, **-**. This switch

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can change the direction of the scan any time. It can be either as an initial direction setting or as a change of direction during an experiment. The direction is shown as either a + or a -, positive or negative going respectively, on the **Scan Rate** display.

4.1.1.8 GAIN nA/V

This switch implies two functions to the CV-37 user; the current to voltage conversion and the gain or amplification. It is important to note and remember that the current generated by the electrochemical reaction at the electrode surface is converted to a voltage and at the same time the signal is amplified for display on a recording device. The **GAIN nA/V** control is a 12 position switch and the current-to-voltage conversion is in nanoamperes per volt (nA/V) of output. The actual current is shown in the display when the **I Out** function is selected. Note the value read in the display is not the voltage at the rear panel output jacks but the actual current in the appropriate units.

4.1.1.9 FUNCTION

The **FUNCTION** switch can be thought of as both an operation and a control switch. When the electrodes (cell) are connected to the CV-37 electronics and the experiment is to begin, this switch will start the potential scanning function by turning this rotary switch to the **SCAN** position. It will stop and hold a potential by turning to the **HOLD** position. The initial potential conditions can be reestablished by turning to the **E1** position or the potential can be stepped from an initial **E1** value to a second or final, **E2**, value by turning the switch to **E2**. If the cell is disconnected, the **E1** and **E2** potential values can be adjusted and fixed. These are adjusted using the **INITIAL E** knobs and choosing either **E1** or **E2** whichever needs changing or monitoring, turning the display to read **App E**, and then turning the respected **E1** or **E2** knob of the **INITIAL E** switch.

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4.1.1.10 CELL MODE

This operational switch turns the cell on and off i.e. rotating the knob to **CELL** connects the electrodes to the CV-37 electronics, and turning the knob to **STBY** or standby disconnects the cell from the electronics. The **TEST POSITION** connects an internal "dummy cell" to the electronics. This is used for testing just the electronics of the unit. Section 5.2 will discuss this function in greater detail. The ΔE function is used for potentiometric measurements i.e. measuring equilibrium or rest potentials. When the knob is in this position, the potential between the reference electrode and the working (or indicating) electrodes is displayed.

4.1.2 Rear Panel

The rear panel is used primarily for connection to external devices for input, recording, and power. This section describes the functions of the components found on the rear panel of the CV-37.

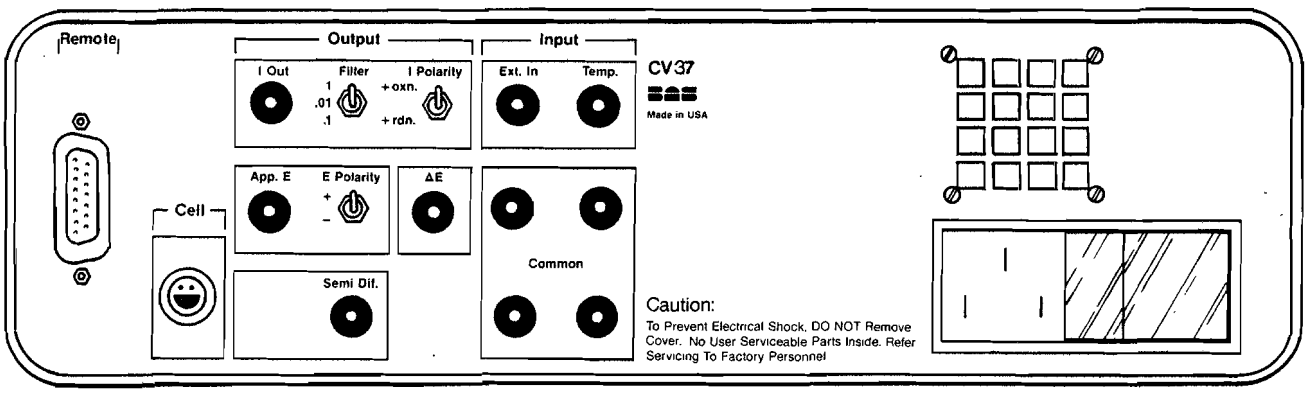


Figure 4.2
Rear Panel of CV-37

4.1.2.1 Cell

This is the mounted part of the cell connecting cable. The cell connecting cable connector simply is pushed in to

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this fitting to make contact between the cell leads or C-1 Cell Stand and the electronics. When inserting this connector make certain of the correct orientation to prevent damage. See section 3.2.1 for additional installation instructions.

4.1.2.2 Output

There are multiple outputs available on the CV-37 unit. The current output, **I Out** with the choice of electronic filtering and polarity, the applied potential, **App. E**, with choice of polarity, the equilibrium or rest potential, **ΔE**, and the semi-differential of the output current, **Semi Dif.** are all available through the rear panel jacks. The choice of output will depend on the particular electrochemical experiment and the response desired. The use and operation is very simple. The output is chosen by plugging the recorder cables into the appropriate jack. Complete the circuit by plugging another plug into one of the common jacks. Note any or all outputs can be monitored simultaneously from this unit.

I Out is the voltage from the current-to-voltage converter and gain control on the front panel. Again note, this is a voltage that corresponds to a particular amount of current being generated by the electrochemical reaction. The conversion is the gain setting on the front panel.

The **Filter** is a passive RC network with time constants of .1, 0.01, and 1 seconds. The amount of electronic dampening chosen by this three position toggle switch depends on the particular electrochemical experiment under investigation. Fast, pulse type experiments usually require a fast (small) time constant whereas slow scanning or DC experiments the longer (large) time constants would be more applicable.

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I Polarity simply allows the user to change the polarity of the output current signal. The **+ Oxn.** is a positive going signal for an oxidation reaction occurring at the working electrode; **+ Rdn.** is a positive going signal for a reduction reaction.

App. E monitors the actual applied potential and is commonly used as the X-axis on the conventional I vs E plots on X-Y recorders. The **E Polarity** toggle switch allows for the choice of output polarities from the **App.E** jack. The **+** position is normal polarity and when toggled to the **-** position the output is inverted.

E is the output of the corrosion potential between the working and reference electrode. Note the reference electrode is at ground.

SEMI DIF is the semi-differential output of the current response from this unit. The use of this signal conditioning will, many times, help resolve closely overlying peaks and eliminate some noise from, for example, a voltammetric experiment. The amount of filtering and the polarity can be changed using the **Filter** toggle switch and the **I Polarity** toggle switch.

4.1.2.3 Input

There are two jacks in the input section of the rear panel. One, the **Ext. In** plug, is used to connect an external voltage to be summed with the potential on the potentiostat. The other, **Temp.**, monitors a voltage that corresponds to the temperature at a liquid probe. This voltage signal must be from the BAS LC-22A Temperature Controller.

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4.1.2.4 Power Cord

The power cord connector allows the unit to be used with multiple input voltages (100 V, 120 V, 220 V, 240 V) as well as providing power line filtering. The female end of the power cord is simply inserted into the power cord connector making certain the pins are properly aligned. See Section 3.1 for additional information on the power cord connection and requirements.

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Section 5.0 Maintenance and Service

This section describes some general cautions, maintenance points, electronic troubleshooting, and the procedure for obtaining service.

5.1 Cautions and General Maintenance

This is a very rugged instrument and with proper care should give years of service. Following is a brief list of cautions and general maintenance considerations that will extend the lifetime of the instrument.

Follow customary, good laboratory practices.

Clean all spills, especially salt solutions, from on or near the cabinet immediately.

Avoid placing unit in a corrosive atmosphere.

Make sure the CELL is off i.e. the **CELL MODE** switch in **STBY** position whenever making cell changes.

Avoid dropping, shaking, and other forms of mechanical abuse since this could cause loosening of components or subassemblies.

5.2 Electronic Troubleshooting

This section specifically describes procedures to isolate suspected electronic failure. Each major section of the instrument will be tested. The unit has a built in circuit, a dummy cell, to assist in testing procedures described below.

Equipment required:

Voltmeter (DVM, 3 1/2 digit is sufficient)

Stop Watch (optional)

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Initial setup conditions:

Plug DVM into **App. E** output jacks.

Rear Panel.

App E polarity to "+" position.

Filter to "0.01"position.

I Out polarity to "+ oxn" position.

Front Panel

DISPLAY to monitor **App E**

CELL MODE in **STBY** position

Potentiostat test:

Turn **FUNCTION** knob to **E1** position

Turn **INITIAL E, E1** knob both fully clockwise and counterclockwise. The LED display and DVM should monitor a range of +5.00 to -5.00 V. The difference between the display and the DVM values should be ±1% of the value.

Turn **FUNCTION** to **E2** and, again, manually scan the potential range of **E2** by turning the **INITIAL E, E2** knob fully clockwise and counterclockwise.

Before proceeding adjust **E2** to -1.00 V and then turn **FUNCTION** knob to **E1** and adjust **E1** to +1.00 V.

Current-to-Voltage converter:

Connect DVM to I Out jack in rear panel.

Turn **GAIN nA/V** to **1000** (lowest gain setting) position.

Turn **CELL MODE** to **TEST** position.

Table 5.1

Troubleshooting Current-to-Voltage Converter (I Out)

App E (E1)	GAIN (nA/V)	I Output (V ± 2%)
+1.00	1000	0.10 (±5%)
+1.00	500	0.20
+1.00	200	0.50
+1.00	100	1.0
+1.00	50	2.0
+1.00	20	5.0
+1.00	10	10.0
+0.10*	10	1.0
+0.10*	5.0	2.0
0.10*	2.0	5.0
0.10*	1.0	10.0
+0.020*	1.0	2.0 ± 10%
+0.020*	0.5	4.0 ± 10%
+0.020*	0.2	10.0 ± 10%

*These should be adjusted using the DVM because of the +10 mV resolution of the DISPLAY value.

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Check the current-to-voltage converter by comparing the values from the DVM to those shown in Table 5.1 for the various **GAIN** and **App E.**

Semi-Differential test:

Turn the **GAIN** to **10**

Turn **FUNCTION** knob to **E2** (which should be -1.00 V).

The **Semi. Dif.** should read 1.1 V on the DVM (this reading may take a while to settle).

Filter:

Move the **DVM** to the **I out jack** and note the meter reading $\approx$ -10 V.

Flip the **Filter** toggle switch on the rear panel to **0.1** seconds; the DVM value will change but return to the previous reading.

Flip this switch to **1.0** seconds, again, the value should change but return to the previous reading.

Return the toggle to **0.01** second position.

I Out Polarity:

Flip **I Polarity** to **+ rdn** and the output should change polarity from -10.00 V to +10.00 (within $\pm$5 mV of previous reading).

Return switch position to **+ oxn** position.

E Output Polarity test:

Move the DVM to the **E out jack** and note the reading

Flip the **E Polarity** switch. You should read the same magnitude and opposite polarity $\pm$1 mV.

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Scan Rate:

Turn **DISPLAY** to **+ Lim**

Adjust to +1.00 V by turning the "+" knob of the **E LIMIT** knob.

Turn **DISPLAY** to **- Lim.**

Adjust to -1.00 V by turning "-" knob of the **E LIMIT** knob.

Turn **DISPLAY** to **Scan Rate.**

Turn **SCAN V/s** knob to **0.10 V/s** position.

Turn **Scan Rate** knob until 100.0 mV/s is displayed in readout.

Turn **DISPLAY** to **App E** position.

Turn **FUNCTION** knob to **SCAN** position.

The **App. E** should be changing with time and should not be going beyond the ± 1.00 V limits. Toggle the **DIRECTION** switch a few times to make certain the scan direction changes appropriately.

Turn the **FUNCTION** knob from **SCAN** to **E1** position.

Adjust **E1** to + 1.00 V.

Toggle **DIRECTION** switch to **NEG.**

Check the actual scan rate by simultaneously turning the **FUNCTION** knob to the **SCAN** position and starting a stop watch. Monitor the App E in the display and as the App E value crosses 0.00 V, stop the watch. This time should be approximately 100 seconds i.e. 1.00 V scan range at 10 mV/s rate = 100 seconds.

Turn **FUNCTION** knob to the **HOLD** position.

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Section 5.3 Service Procedure

There are no user serviceable parts in this unit and all service requests should be referred to BAS service personnel. In certain cases, BAS will provide electronic schematics and service procedures to qualified electronic maintenance facilities but only upon written request and then only on the approval of the Service Coordinator.

If a problem arises and appears equipment oriented, call BAS at (317) 463-4527 and ask for Customer Service. The operator will route you to your Sales Representative. Be prepared with the following information;

1. Model Number of the instrument
2. Serial Number of the instrument
3. When purchased
4. Description of the problem in detail, including "testing" information.

If the Sales Representative can not resolve your problems, he/she will take your name, institution, address, and phone number and will forward the above information to the Service Coordinator. The Service Coordinator will work with you to get a satisfactory answer to your service problem. He/she has specific technical personnel available to him/her to solve all problems, i.e. electronic problems will be directed toward to electronic engineers, application or chemical problems to staff chemists, etc. If required, the technical person will call you back and further discuss your problem.

If an item needs to be returned for service, repair, or in house investigation, the Service Coordinator will assign you a Customer Service Number (CS#). Do not return any goods without a CS#. It identifies you, your problem,

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what actions have already been taken. A CS# quickly and efficiently moves your service problem and equipment through the service protocol. Again, do not return any item without a CS#. It will not be accepted!

All correspondence and shipments should be sent to

Bioanalytical Systems Inc.
2701 Kent Avenue
Purdue Research Park
West Lafayette, IN 47906 USA

Items being returned for service must clearly show the CS# on the outside of the package.

Service will normally be completed and returned to you within 10 working days.

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Section 6.0 Sample Voltammetric Experiments

This section describes a series of simple voltammetric experiments to familiarize the user with the operation of the CV-37 instrument. You must do these experiments to obtain a working knowledge of the voltammetry controller! When these sample experiments have been completed, you should have sufficient operational know-how to very rapidly adjust the instrument to the parameters required for your in-vivo experiment.

You will need the following to perform these experiments with the CV-37. The materials for these experiments include:

1. Prepared sample solution (0.1 mM Dopamine in 0.1 M perchloric acid)
2. Voltammetry cell with an electrode holder
3. Micro Working electrode
4. Auxiliary electrode e.g. Platinum wire mounted in voltammetry cell top
5. Reference electrode: e.g. RE-1 Ag/AgCl
6. Sample Vial
7. X-Y Recorder
8. Recorder paper

Before proceeding with the experiments, make certain the power cord has been plugged into both the instrument and an appropriate grounded wall socket. This unit should be connected to an X-Y recorder using the leads provided; **Output** connected to the Y-axis on the recorder

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and **App. E** (-,E Polarity) connected to the X-axis. Note, when the recorder is connected in this manner, the resulting voltammogram will follow usual electrochemical sign convention. (See Section 3 for additional information on connecting power cord, cell lead, and recorder leads.) Also, to obtain voltammograms similar to the examples shown below, the output filter time constant must be in the 0.01 position.

6.1 Preparation of Electrochemical Cell and Sample Test Solution

A. Place dopamine solution into sample vial.

B. Place Auxiliary and Reference electrodes into sample vial.

C. Prepare the Micro Working electrode and place into cell.

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D. Connect cell leads to electrodes; black lead to carbon paste working electrode, red lead to platinum auxiliary electrode (mounted gold pin in voltammetry cell top), and white (or clear) lead to reference electrode.

E. Make certain the **CELL MODE** function switch is in the **STBY** position and then turn **POWER** switch to **ON** (up) position. A red indicator light above switch will be illuminated when power is applied to the instrument.

6.2 Control Switches Familiarization: Adjusting Initial Experimental Conditions

A. Adjust + and - LIMITS (scan limits) to +0.90V and -0.20V respectively. To adjust the + LIMIT:

1. Turn **DISPLAY** knob until the **+LIM** function is shown in the status area of the display window.

2. Turn the **E LIMIT, +,** (inside) knob until the LED display shows 0.90V. (The **V** symbol should also be illuminated in the status area of the display window.)

3. Turn the **DISPLAY** knob, again, until the **-LIM** function is shown in the status area of the display window.

4. Turn the **E LIMIT, -,** (outside) knob until the LED display shows -0.20V.

B. Adjust INITIAL E, E1 to +0.00 ± 0.005V.

1. Turn the **DISPLAY** knob unitl **App. E** is shown in the display window.

2. Turn the **FUNCTION** switch to the **E1** position.

3. Turn the **INITIAL E, E1,** (inside) knob until 0.00 is shown by LED display.

C Adjust Scan Rate to 20 mV/sec. in the positive going direction.

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1. Turn **DISPLAY** knob until **SCAN RATE** is shown on the display window.

2. Turn the **SCAN V/S** switch to the 0.10 position.

3. Turn the **SCAN RATE** knob until 20.0 is shown in the display. It can be + or -.

4. Toggle the **DIRECTION** switch up and down, **POS** and **NEG** respectively, and notice how the direction is shown on the LED display; nothing for positive direction, - for negative direction. Leave the direction positive going.

D. Adjust the signal gain to 1000 nA/V

1. Turn the **GAIN nA/V** knob to the 1000 position. This number i.e. 1000 is the "conversion factor" between the output voltage measured at the output I **Out** jack and the current actually will be generated at the working electrode. For example if there is an output of 0.4V, this corresponds to 400 nA i.e. 0.4V x 1000 nA/V = 400 nA. If there were 6.0 volts of output, the corresponding current would be 6.0 V x 1000 nA/V = 6000 nA or 6.0 uA.

6.3 Experiment 1: Generating a Cyclic Voltammogram

Before continuing with this experiment, make sure all leads to the recorder are properly connected, the power to the recorder is ON and the range switches on the recorder are set on 0.1V/cm on both the X and Y scales, and the cell lead wires are connected to the three electrodes in the test solution following the proper color code.

Place the paper on the X-Y recorder (either the paper provided with the instrument of any appropriate paper to fit your X-Y recorder) and using the zero controls on the recorder, position the pen at the (0, 0) point of the X and Y axis.

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The first set of experiments are designed to illustrate the effects on the voltammogram and what the instrument does if the scan is allowed to proceed to the "+" or "-" limits, i.e. generating a cyclic voltammogram.

The conditions are as described above. Once again they are:

Solution/Cell

0.1 mM Dopamine solution (0.1 M $HClO_4$)
Micro Working Electrode (Area 2mm^2)
Reference Electrode, e.g. Ag/AgCl

Instrument

+LIM = 0.90V
-LIM = 0.20V
E1 = 0.00V
SCAN RATE = 20.0 mV/s
GAIN nA/V = 500

Experiment

1. Turn the **DISPLAY** knob until the **App. E** function is shown in the display window.

2. Make certain there is not any air bubbles under the working electrode or the reference electrode in the cell.

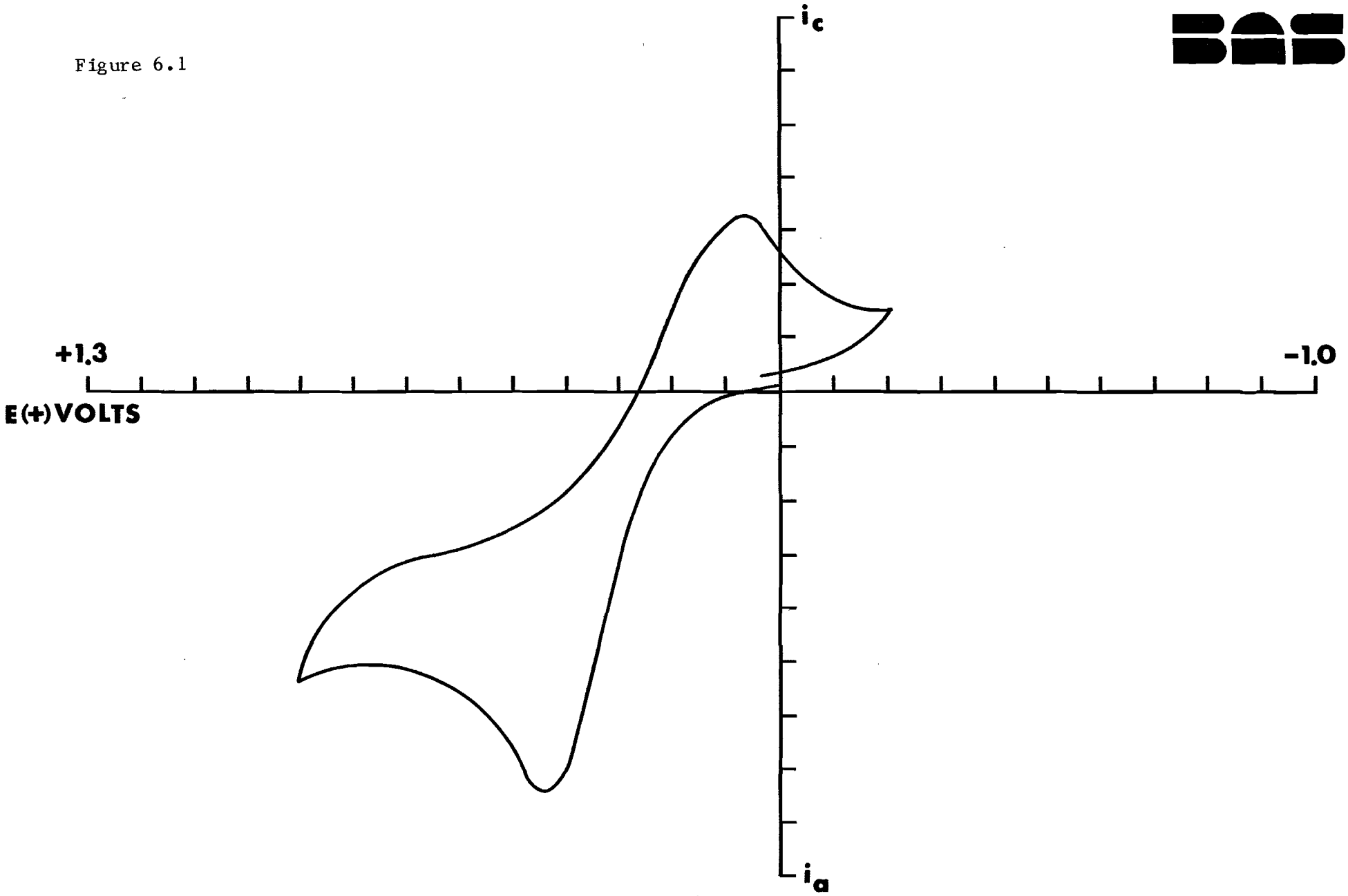
3. Place the pen down on the paper.

4. Turn the **CELL MODE** switch to the **CELL** position. This turns the cell on i.e. connects the working electrode to the current measuring circuitry. There should be a small "spike" on the chart paper corresponding to the cell being turned on.

5. Turn the **FUNCTION** switch to **HOLD** position and then to the **SCAN** position. The pen will begin to move to the left on the XY recorder and the readout on the display will change, continuously, to more positive values. When the potential reaches +0.90V the scan direction changes from positive to negative and the applied potential now begins decreasing. As the

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Figure 6.1



DATE: 2-21-84	FILE #:	TRACED BY:
SAMPLE: DOPAMINE		
CONC: 0.1mM	SCAN RATE: 20mV/sec.	
SENSIT: 500nA	W. ETRODE: micro	REF: Ag/AgCl
MEDIUM: 0.1M $HClO_4$		

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potential reaches approximately +0.20V, the current (anodic) begins increasing rapidly due to the oxidation of the dopamine to the quinone. It will reach a maxima at about +0.40V and then begin to decrease. This gradual decrease continues until the **+LIM** potential is reached. Again, this is the scan direction switching potential. So the scan now continues in a negative going direction, the anodic (oxidation) continues to decrease, until approximately +0.25V where reduction of the generated quinone begins to occur. This cathodic current also reaches a maxima at approximately +0.10V.

6. As the potential scan reverses and approaches zero, turn the **FUNCTION** switch from the **SCAN** to the **HOLD** position.

7. Turn the **CELL MODE** switch to the **STBY** position to disconnect the electronics from the cell.

The resultant "cyclic" voltammogram is shown in Figure 6.1.

6.4 Experiment 2: Generating a Linear Sweep Voltammogram and Response versus Scan Rate

This second experiment is designed to show the user how to generate Linear Sweep Voltammograms and how the response is effected by charges in the scan rate.

The single sweep or linear sweep potential wave form is the common one used for the in-vivo experiment.

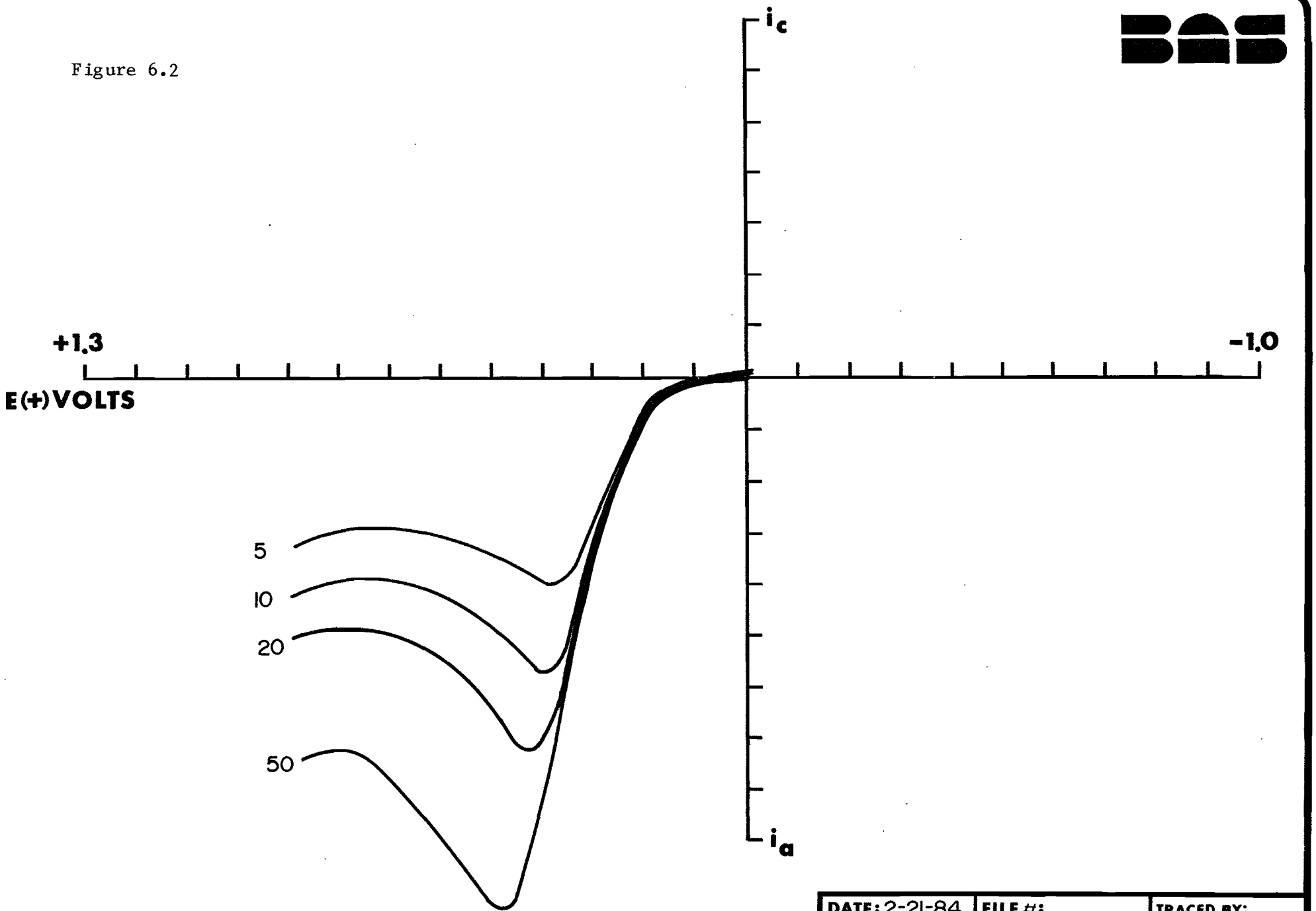
Initial Experimental Conditions:

INITIAL E, E1 = +0.10V
+LIM = +1.90V
-LIM = -0.00V
SCAN RATE = +5.00 mV/s (The is the variable in
DIRECTION = POS. (Toggle) this experiment)
GAIN = 500 nA/V
DISPLAY - monitoring App. E

Before continuing check:
CELL MODE switch in STBY position.
FUNCTION switch in either E1 or HOLD position.=

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Figure 6.2



DATE: 2-21-84	FILE #:	TRACED BY:
SAMPLE: DOPAMINE		
CONC: 0.1mM	SCAN RATE: 20mV/sec.	
SENSIT: 500nA	W. ETRODE: micro	REF: Ag/AgCl
MEDIUM: 0.1M $HClO_4$		

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Place new piece of paper in recorder with recorded zeroed at axis' origins. Note the output from the App. E jack is +0.00V; Adjust accordingly.

Experiment:

1. Put the recorder pen down, turn the cell on, i.e. **STBY** position to the **CELL** position, and turn the **FUNCTION** switch to the **SCAN** position. Wait for current to settle to zero.

2. When the scan reaches approximately +0.90V, stop the scan by turning the **FUNCTION** switch from the **SCAN** to the **HOLD** position.

3. Turn the cell off by turning the **CELL MODE** switch to the **STBY** position. (The pen will return to zero on the Y or current axis.)

4. Lift the pen and turn the **FUNCTION** switch to the E1 position. The pen will return to the +0.00V position on the X or potential axis.

5. To complete this experiment (a set of voltammograms) repeat the experiment but at different scan rates e.g. 10, 20, 50. Typical current responses versus scan rate are shown in Figure 6.2. Note, the increase in peak current as the scan rate is increased. This increase in current is directly proportional to the square root of the scan rate. i.e.

$$i_{peak} = K\, v^{1/2}.$$

Of course, then, a plot of the i peak versus $v^{1/2}$ should yield a straight line of slope K (see equation 2.1).

6.5 Experiment 3. Comparison of Normal and Semidifferential Current Outputs and Quantitation

This experiment is to illustrate the difference between the normal or standard current response and the semidifferential response. The latter has been used by a number of researchers in-vivo electrochemistry area. See the In-Vivo Bibliography for references. The apparent effect is

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that under typical in-vivo experimental conditions, peaks are better defined and backround currents (response) are attenuated; thus, improved resolution and detection limits as compared to the normal output.

Initial Experimental Conditions:

INITIAL E, E1 = +0.00V
+ LIM = +0.90V
- LIM = +0.00V
SCAN RATE = +50 mV/s
DIRECTION = POS. (Toggle)

GAIN = 500 nA/V
CELL MODE switch in STBY position.
FUNCTION switch in E1 or HOLD position.
DISPLAY monitoring App. E

New piece of paper in recorder.

Experiment:

1. Put the recorder pen down, turn the cell on and start the scan.

2. Again as in the previous experiment, when the scan reaches approximately 0.90V stop the scan by turning the **FUNCTION** switch from the **SCAN** position to the **HOLD** position.

3. Turn the cell off.

4. Lift the pen on the recorder and turn the **FUNCTION** switch back to the **E1** position.

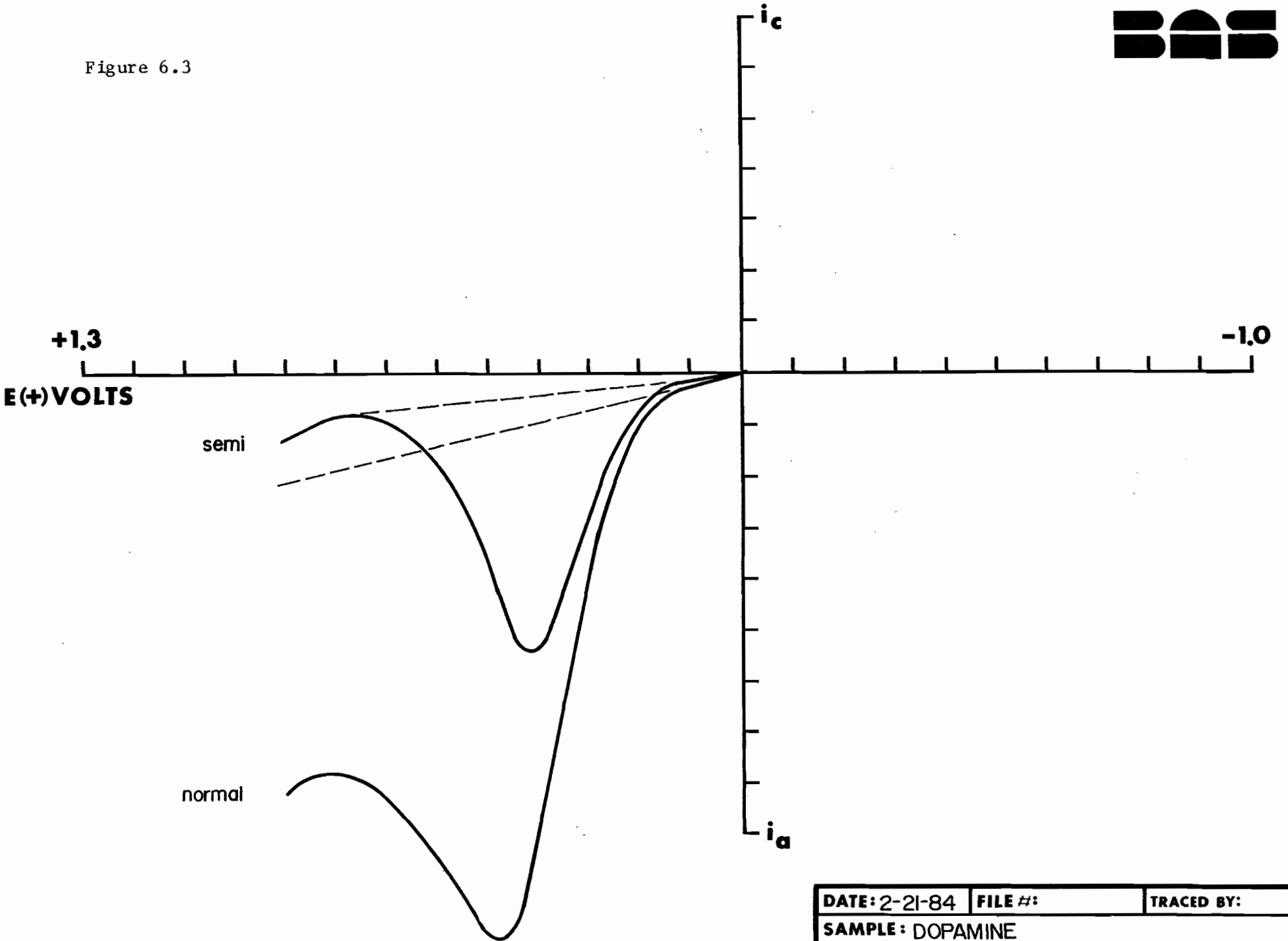
5. Now rerun the experiment, monitor the semidifferential output (i.e. the cable to Y-axis of recorder plugged into the Semidifferential output instead of the **I OUT** output.

A typical response of each is shown in Figure 6.3.

Note that peak heights in both cases are directly related to concentration i.e. i peak = K [R or O] where R and O are the

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Figure 6.3



DATE: 2-21-84	FILE #:	TRACED BY:
SAMPLE: DOPAMINE		
CONC: 0.1mM	SCAN RATE: 20mV/sec.	
SENSIT: 500nA	W. ETRODE: micro	REF: Ag/AgCl
MEDIUM: 0.1 M $HClO_4$		

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reduced or oxidized forms of the electrochemically active species of interest. Peak currents are found by measuring the difference between the maximum current (i.e. the peak height) and an extrapolated baseline. The baseline is extrapolated from the data prior to the point where the Faradic reaction begins. An example is shown by the dashed line in Figure 6.3.

6.6 Experiment 4: Voltammogram using Micro Electrodes

The in vivo experiments are done with electrodes much, much smaller in area than the electrode used for these experiments. The response of micro electrodes i.e. electrodes with diameters of 5-10 microns, are different than larger electrodes i.e. electrodes with diameters of hundreds of microns.

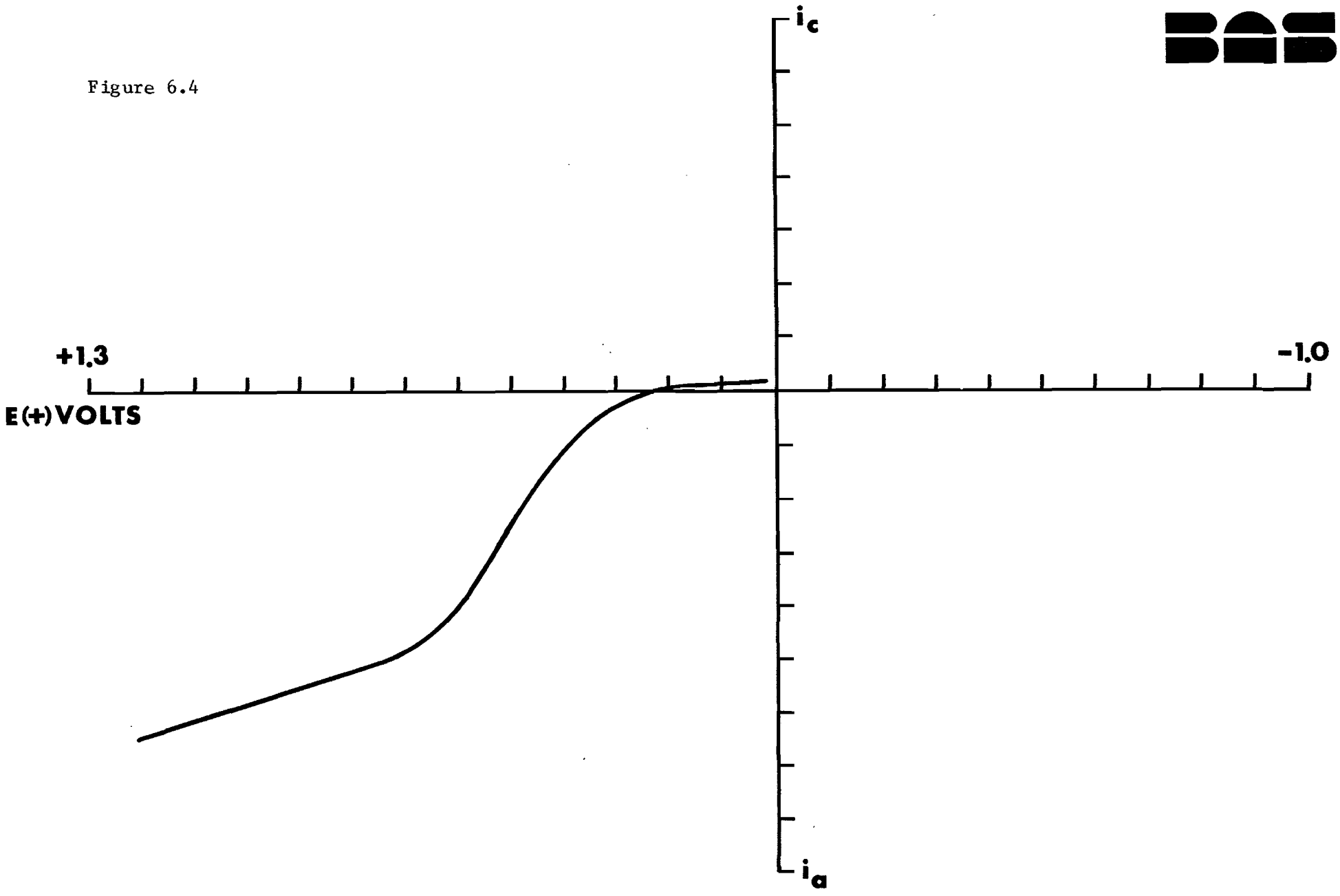
If these small diameter (5-10 um) micro electrodes are going to be used, it is worthwhile to observe some of the differences; at least in the in-vitro (beaker) experiments. Figure 6.4 is a voltammogram under the same conditions as Experiment 3. There are two points worth noting. First, the GAIN is higher since the area of the electrode is smaller and thus currents are smaller and, second, the shape of the curve is sigmoidal rather than having a peak shape. The reason the typical response is not observed is because under these conditions (slow scan rate) the rate of electrolysis is approximately equal to the rate of diffusion to the electrode. A much more thorough discussion of this phenomena is given in a review article by Dr. R. Mark Wightman, "Microvoltammetric Electrodes," Anal. Chem., 53(1981) 1125A-1134A.

6.7 Experiment 5: Potential Step

In some cases it may be advantageous to apply a potential step rather than continuously varying potential waveform.

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Figure 6.4



DATE: 2-21-84	FILE #:	TRACED BY:
SAMPLE: DOPAMINE		
CONC: 0.1mM	SCAN RATE: 5mV/sec.	
SENSIT: 2nA/V	W. ETRODE: micro	REF: Ag/AgCl
MEDIUM: 0.1 M $HClO_4$		

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In general (very general) the reasons may be to improve selectivity (to a limited degree) and, perhaps, it can improve detection limits. To illustrate that a potential step experiment can be done using this instrument, consider the conditions described in Experiments 1-3. The voltammetry indicates that very little reaction occurs at +0.00 V and the reaction is going to rapid completion at approximately +1.00V. Thus it would seem reasonable that if the potential is stepped between these two limits, the resultant difference current should be due to the reaction of the electrochemically active substances only. Stated differently, at the lower potential, the current would be due to unwanted background materials reacting and at the higher potential not only these same unwanted background reactions would be occurring but also the reactions of interest.

Initial Experimental Conditions:

Cell
Micro Working electrode
Reference electrode, e.g. Ag/AgCl
Dopamine solution
Cell (Same Vial)

Instrument
INITIAL E, E2 = +0.10V
INITIAL E, E1 = +1.00V

GAIN = 100 nA/V
CELL MODE switch in STBY position.
FUNCTION switch in E2 position.
DISPLAY monitoring I Out.

In this experiment the response is monitored as a function of time rather than potential so the output from the I Out jack is attached to a Y-T, time base (strip chart) recorder. See section 3 for specifics on how to connect strip chart recorder. Full scale range on the recorder should be 10 Volt. The chart speed should be about 10 cm/min. Zero the recorder in the center of the paper. Also a stopwatch, watch, or clock with second resolution will be required.

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Experiment:

1. Start the recorder.

2. Turn the cell **ON**.

3. Turn the **FUNCTION** switch to the **E1** position for 5-10 seconds and then return to the **E2** position.

4. Repeat the steps from E2 to E1 once every 30 seconds.

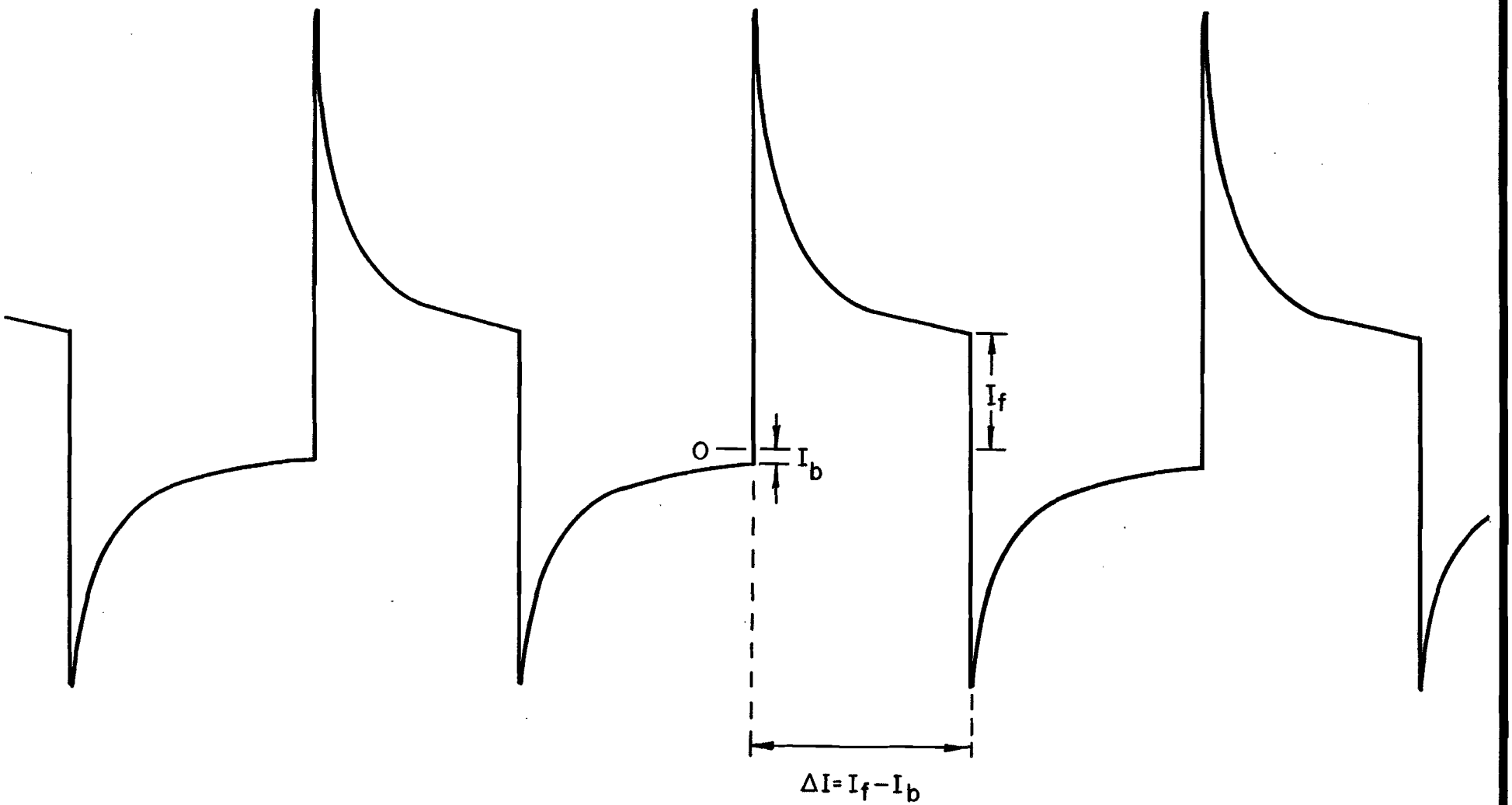
The response is shown in Figure 6.5. Note the method of current measurement and the values used to give the difference measurement.

Repeat the above experiment and leave the pulse width at 30 seconds but repeat the pulse once every two minutes. This response is shown in Figure 6.6.

Since this experiment is obviously quite labor intensive, and there would be considerable chance of manual error in time, it should be controlled with a timing input through the remote jack on the rear panel. A number of timers are available commercially (this could be run by a "computer" but it's a substantial overkill) that would meet the requirements of the CV-37 inputs as described in Section 3 of this manual.

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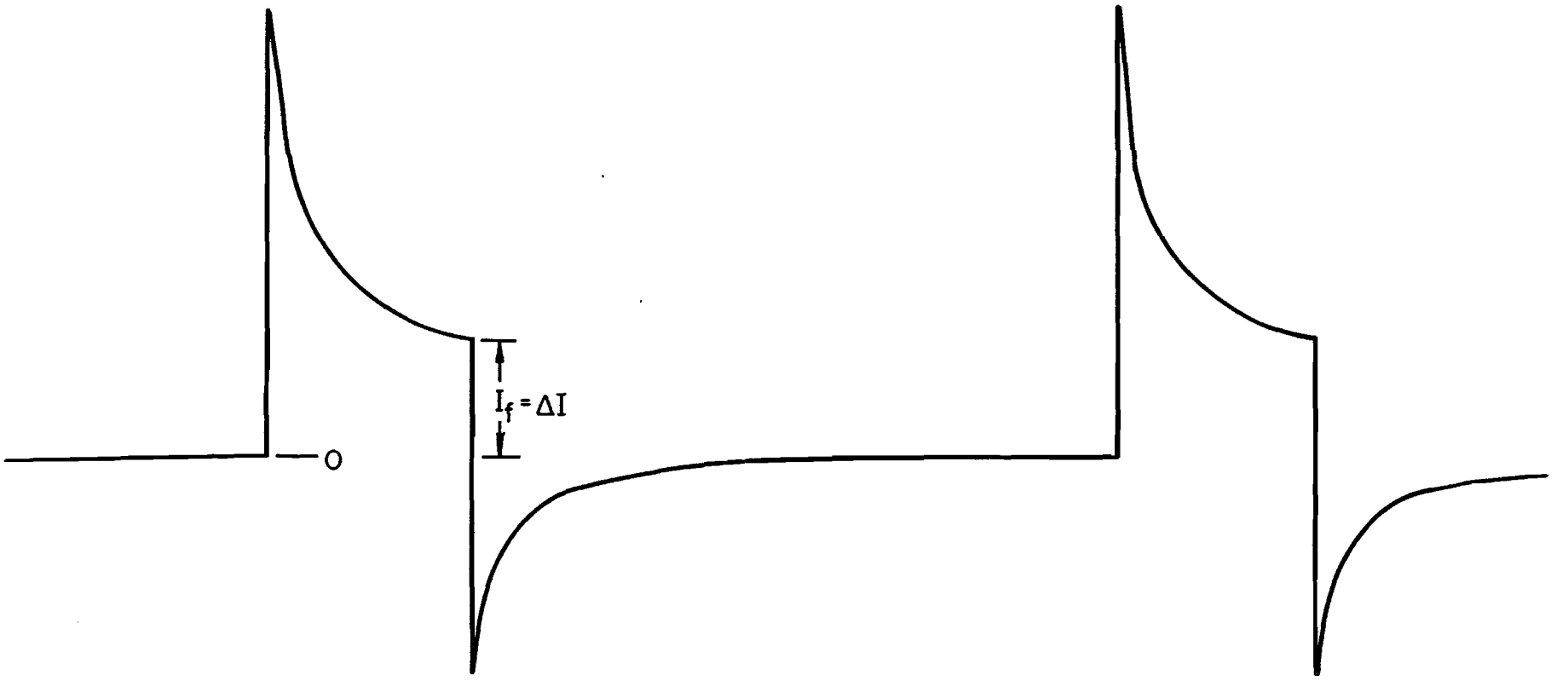
Figure 6.5



DATE: 2-21-84	FILE #:	TRACED BY:
SAMPLE: DOPAMINE		
CONC: 0.1 mM	SCAN RATE:	
SENSIT: 200 nA/V	W. ETRODE: micro	REF: Ag/AgCl
MEDIUM: 0.1 M $HClO_4$		

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Figure 6.6



DATE: 2-21-84	FILE #:	TRACED BY:
SAMPLE: DOPAMINE		
CONC: 0.1 mM	SCAN RATE:	
SENSIT: 200 nA	W. ETRODE: micro	REF: Ag/AgCl
MEDIUM: 0.1 M $HClO_4$		

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