

SECOND QUARTERLY REPORT

21 November 1964 - 20 February 1965

GASEOUS ELECTROLYTES FOR
BATTERIES AND FUEL CELLS

by

S. Nalditch, Principal Investigator

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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1. ABSTRACT

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The detailed approaches and techniques used on this program have been explained in the preceding progress reports. Briefly, the objective is to evaluate the applicability of dense gaseous electrolytic solutions to energy conversion. Ammonia is being used as the gaseous solvent. The experimental studies are being carried out in hermetically sealed glass vessels. Since the gaseous solvents are at pressures of the order of 112 atmospheres, external pressure is used to prevent rupture of the glass.

The cell design has undergone many modifications to reduce or eliminate experimental problems that have arisen on this program. Since the present design appears to satisfy the principal requirements, we have summarized its main features in section 2.

We now have reproducible EMF data at low temperatures (section 3) and preliminary EMF and conductivity data to about 100°C (section 4). Various techniques are being examined in order to extend the data above this temperature. The difficulty is that as the critical region is approached, the solutions become turbulent and the half cells develop open circuit characteristics.

A handwritten signature, possibly reading "Sulthor", is written in dark ink. It consists of a series of fluid, connected strokes, with a long horizontal line extending from the bottom right.

2. CONDUCTIVITY CELLS

The basic approach used on this program has been to contain the dense gaseous electrolytic solutions in hermetically-sealed glass tubes. Since the gaseous pressures exerted by these solutions are of the order of 100 - 200 atmospheres, external pressures greater than the solution pressures are used to keep the cells from rupturing, glass being stronger under compression than under tension.

The usable space inside the pressure vessel is cylindrical, nine inches long by one and one-half inches in diameter. Because of this restrictive geometry, we have chosen to use DC for the conductivity measurements, since capacitive and inductive effects between the various components of the cell may become appreciable if AC is used under such conditions.

A four-electrode cell is being used for these studies, one pair for the measurement of equilibrium EMFs, and the four electrodes for electrolytic conductivities, the two extreme electrodes being used to carry the current and the two intermediate electrodes to measure EMFs for conductivity measurements. The cylindrical tubular section between the two EMF-measuring electrodes is called the "conductivity tube" in the following discussion. The factors on which the cell design (Fig. 1) is based are as follows:

1. The cell has been designed to produce a uniform current density in the conductivity tube. When this condition is satisfied, the cell constant K is equal to L/A , the ratio of length to internal cross-section area of the conductivity tube.
2. The potential difference between the two ends of the conductivity tube is measured through narrow slit apertures perpendicular to and encompassing the entire circumferences of the conductivity tube at each end of this tube. This geometry has been chosen instead of the

small circular hole as in a Luggin electrode. One reason is that if there are any non-uniformities in the current density electrolyte, or EMF distribution, use of the circular slit should average them out. That is, we scan or sample the EMF over a large region instead of at a small locality. Hence, effects of non-uniformities in the solution will be reduced. Secondly, for a given scanning area, the slit can be made narrower than a circular hole, therefore the potential drop across the width of the slit is less than across the hole. Under this condition, there is less penetration of the current into the EMF-electrode measuring region.

3. The cell can be easily pre-fabricated to within one percent of any desired cell constant.
4. The areas of the amalgam-solution interface are maximal.

The characteristics of the cells have been measured by several different techniques, the three cell parameters, the length, L , the effective cross sectional area, A , and/or the cell constant, K , having been deduced as follows:

2.1 Areas

The geometrical dimensions of the conductivity tube have been measured with calipers before final assembly by the glassblower. From those measurements of the length and the inner diameter, the cell constant K is calculated as

$$K = L/A \quad (1)$$

The numerical values of A , L , and K are tabulated in Tables 1, 2 and 3, respectively.

With the cell, except for the freeze cup, completely assembled, cylindrical rods were inserted into the cell (Fig. 2). The cross sectional area of

each rod was measured with a micrometer. The voltage, V (B-C), was measured as a function of current, I (A-D), for the cell with one rod in place and the slope, S_r , of voltage versus current, determined graphically. With known cross sectional area of the rod serving as an additional parameter, the product of σ , specific conductivity of the electrolyte, and L , can be dropped from the equations.

The effective cross-sectional area, now called A_0 , may be determined by an independent technique in which $E-I$ slopes are determined without and with a rod of known diameter placed inside the cell. With $S_0 = dE/dI$ and S_r , the corresponding slope with the rod in place, then

$$S_0 = \sigma L/A_0$$

and

$$S_r = \sigma L/(A_0 - A_r)$$

where A_r is the cross-sectional area of the inserted rod. The areas are then calculated from:

$$A_0 = \frac{A_r}{1 - \frac{S_0}{S_r}} \quad (2)$$

The respective data for mercury and KCl solutions are shown in Figs. 3 and 4.

Data obtained with both KCl solutions and mercury are included in Table 1. The agreement between the calipers and the rods in KCl and mercury is only fair. It was found later that the resistor used for measuring the current was overheating.

TABLE 1

Internal Effective Cross-Sectional Area of Conductivity Cell No. 3

	$A_o, \text{ cm}^2$
Calipers	0.278
Rods	
KCl No. 3	0.272
Hg No. 6	<u>0.289</u>
Ave.	0.280 ± 0.006

2.2 Lengths

After the cell was fabricated, the length was remeasured with a cathetometer. This measurement (Table 2) agrees well with the caliper measurement; however, because of the distortions of the optical paths in the regions of ring seals, it cannot be used as a reliable method of measurement.

In addition to the direct measurements of length with cathetometer and caliper, a rod technique was used to measure the effective length of the conductivity tube. The rod was inserted into the cell, and its position varied. The voltage to current ratio was measured as a function of the rod position. The slope, S_x , of the curve of V/I versus position was used to calculate the length of the conductivity tube. The data are plotted in Fig. 5.

Let the length of penetration of the rod into the conductivity tube be x . The resistance, R_x , in this tube consists of two series resistances, one from zero to x with the center occupied by the rod and from x to L with the center clear. Then

$$R_x = \rho \frac{x}{A_o - A_r} + \rho \frac{L-x}{A_o}$$
$$S_x = \frac{dR_x}{dx} = \rho \frac{A_r}{A_o(A_o - A_r)}$$

Since $R_o = \rho \frac{L}{A_o}$, when we solve for L , the specific resistivity drops out.

The lengths are calculated from

$$L = \frac{R_o}{S_x} \frac{A_r}{A_o - A_r} \quad (3)$$

where R_o is the resistance of the conductivity tube without rod.

It is seen in Table 2 that agreement is fairly good between the rod and the caliper technique.

TABLE 2

Lengths

	L, cm
Calipers	4.19
Cathetometer	4.22
Rods (S_x); $L = \frac{R_o}{S_x} \frac{A_r}{A_o - A_r}$, average A_o , micrometer A_r	

KCl Solution No. 1, Rod No. 3, $L = 4.25$

Average 4.22 ± 0.02

2.3 Cell Constant K

In addition to the caliper measurements, a second method (generally used in electrochemistry) is to use the specific conductivity of KCl to establish K. Two different KCl solutions were used and a single measurement made on each solution. The cell constant is calculated from $K = R \sigma$. In addition, with the first solution the slope, S_o , of voltage versus current was measured. The cell constant was calculated from: $K = S_o \sigma$. The agreement with the caliper results (Table 3) was poor.

A similar measurement was carried out with mercury. Again, agreement was poor. Here, however, it was noticed that the resistor was undoubtedly overheated.

Two different rod techniques were used to measure the cell constant. In one, the rod was inserted all the way into the cell and the cell constant was calculated from S_r , the slope of voltage versus current with the rod inserted, σ , the specific conductivity, A_o , and A_r . Since

$$S_r = R_r = \rho \frac{L}{A_o - A_r},$$

and

$$K = \frac{L}{A_o},$$

then

$$K = S_r \sigma \left(\frac{A_o - A_r}{A_o} \right) \quad (4)$$

The second rod technique was used with KCl with the rod at various positions. The cell constant was calculated from:

$$K = \frac{R_o}{S_x} \frac{A_o A_r}{A_o (A_o - A_r)} \quad (5)$$

TABLE 3

Determination of the Cell Constant, K

1. Calipers	$K = \frac{L}{A} = 15.07$
2. Average L, average A (Tables 1, 2)	$K = \frac{L}{A} = 15.07$
3. $K = R\sigma$ (R measured at a single voltage)	
a. KCl solution No. 1	14.32
b. KCl solution No. 2	14.38
4. $K = S_o\sigma$, where $S_o = \partial V/\partial l$	
a. Hg	$K = 15.95$
b. KCl solution No. 1	14.00
5. Rods inserted all the way in, $K = S_r\sigma \frac{A_o - A_r}{A_o}$, average A_o , micrometer A_r	
a. Hg Rod No. 6	$K = 14.83$
b. KCl Rod No. 3	$K = 14.35$
6. Rods at various positions; $K = \frac{R_o}{S_x} \frac{A_r}{A_o(A_o - A_r)}$, average A_o , micrometer A_r	
KCl Rod No. 3	$K = 15.32$

3. LOW TEMPERATURE STABILITY AND REPRODUCIBILITY MEASUREMENTS

Efforts have been directed toward removing the sources of instabilities encountered in the cells in the first quarter. Measurements have been made on a zinc-cadmium cell (No. 12) from -74 to -11°C . These measurements have been restricted to the lower temperature, where the experimental techniques are simpler, so that the studies could be concentrated on the instabilities.

This cell had excellent stability. Measurements made on three different days at a number of temperatures have an average mean deviation of 0.3 millivolts at each temperature. The cell, which had no expansion bulb, was almost completely filled with NH_3 . Zinc amalgam and ZnCl_2 were put in electrode compartments A and D and cadmium amalgam and CdCl_2 in the other two cell compartments. Since the cell was almost completely filled, it could be used only at lower temperatures, there being only a little volume for expansion.

For the measurements, the cell was placed in an insulated thermostated compartment which could be regulated from -80° to well above room temperature.

Contact was made to one zinc and one cadmium electrode with alligator clips. A Leeds and Northrop Type K-3 potentiometer was used for the potential measurements and the temperature was monitored with a copper-constantan thermocouple which was read with a Hewlett-Packard VTVM.

EMFs of cell No. 12 were measured over the temperature range of -74 to -11°C . The measurements in Table 4 were made on the second day (the first day being taken as the day when the cell was filled). The stability on the second day was not as good as in the succeeding days. Possibly more time was needed for the ammonium chloride solution to become uniform. For this reason, the data of the second day are omitted from further consideration. Three additional sets of measurements were made on the third, eighth and ninth days, which are tabulated in Tables 5, 6, and 7.

Data for each temperature from Tables 5, 6 and 7 were averaged, and the results recorded in Table 8 and plotted in Figure 6. The plotted points represent the mean values obtained for a given temperature. From 2 to 6 measurements are therefore represented by each data point, except for the non-equilibrium point at -42° . It will be noted from the tables that agreement to within a few tenths of a millivolt was usually attained, thus lending considerable confidence to the measurements. It is possible that the small residual scatter of data is due to temperature variation rather than to EMF variations. With the particular thermostat being used, it is difficult to hold the temperature to much better than a degree for prolonged periods of time.

One question that was raised earlier was whether freezing of the amalgams might affect the results. The two amalgams used differ in that cadmium forms a solid solution with mercury while zinc does not. The curve in Figure 6 shows that a transition occurs at about -39°C . It is not certain whether it is caused by the phase separation occurring on freezing of the zinc amalgam, since any freezing, with or without phase separation, will in general cause such a transition. It is interesting, nevertheless, to note that the transition does occur in the neighborhood of the melting point of mercury, -38.7°C , and of the mercury-zinc eutectic, -41.6° .

The reason for the negative slope at the higher temperature branch of our curve, instead of the positive slope obtained by Yost, may be due to variable compositions of the zinc and cadmium amalgams. An inspection of the phase diagrams for the cadmium-mercury and mercury-zinc systems, reveals that with our amalgam compositions (about 1 to 2 weight percent for each amalgam) there was probably a variable amalgam composition over the entire branch of our curve above the freezing point of mercury.

When the temperature was lowered from -37 to -42°C , a large increase in the EMF was observed. It persisted for about five minutes, then slowly dropped. At the end of about half an hour, it had steadied down to a lower value in accord with the other data. The initial high value is recorded in Figure 6 with a plus. It may be a metastable point corresponding to super-cooled liquid amalgam.

TABLE 4
EMF OF CELL NO. 12
AS A FUNCTION OF TEMPERATURE
MEASURED ON THE SECOND DAY

<u>T, °C</u>	<u>EMF, VOLTS</u>
-27	0.4416
-33	0.4407
-33	0.4405
-33	0.4405
-42	0.4407
-58	0.4219
-74	0.4145
-44	0.4336
-44	0.4346
-44	0.4353
-44	0.4353
-37	0.4396
-37	0.4399
-33	0.4345
-27	0.4365
-27	0.4367
-15	0.4321
-15	0.4323
-27	0.4369
-27	0.4367
-33	0.4392
-33	0.4395
-37	0.4399
-37	0.4363

TABLE 5
EMF OF CELL NO. 12
MEASURED ON THE THIRD DAY

<u>T, °C</u>	<u>EMF, VOLTS</u>
-15	0.4229
-15	0.4226
-27	0.4287
-27	0.4286
-33	0.4310
-33	0.4309
-37	0.4373
-37	0.4381

TABLE 6
EMF OF CELL NO. 12
MEASURED ON THE EIGHTH DAY

<u>T, °C</u>	<u>EMF, VOLTS</u>
-11	0.4231
-11	0.4218
-11	0.4221
-20	0.4267
-20	0.4263
-25	0.4277
-25	0.4275
-27	0.4284
-27	0.4286

TABLE 7
EMF OF CELL NO. 12
MEASURED ON THE NINTH DAY

<u>T, °C</u>	<u>EMF, VOLTS</u>
-15	0.4258
-15	0.4254
-27	0.4283
-27	0.4291
-33	0.4328
-33	0.4328
-37	0.4377
-37	0.4382
-42	0.4561*
-42	0.4352
-44	0.4322
-44	0.4326
-58	0.4202
-58	0.4204
-74	0.4101
-74	0.4101
-58	0.4194
-58	0.4194
-44	0.4321
-44	0.4324
-42	0.4353
-42	0.4351
-37	0.4374**
-37	0.4322***
-15	0.4251
-15	0.4257
-37	0.4381
-37	0.4377

* This point was unstable. The following equilibrium value was reached after about 30 minutes.

** These two points were unstable. A steady value was reached only after the cell had been warmed to -15°.

TABLE 8
MEAN EMF VALUES OF
DATA IN TABLES 5, 6 AND 7

<u>T, °C</u>	<u>No. of Runs</u>	<u>EMF, Volts</u>
-11	2	0.4220 $\pm$ 0.0001
-15	6	0.4246 $\pm$ 0.0012
-20	2	0.4265 $\pm$ 0.0002
-25	2	0.4276 $\pm$ 0.0001
-27	6	0.4286 $\pm$ 0.0002
-33	4	0.4319 $\pm$ 0.0009
-37	6	0.4378 $\pm$ 0.0003
-42	3	0.4352 $\pm$ 0.0001
-44	4	0.4323 $\pm$ 0.0002
-58	4	0.4198 $\pm$ 0.0004
-74	2	0.4101 $\pm$ 0.0000

4. DATA AT ELEVATED TEMPERATURES

The principal efforts in this past quarter have been directed toward extending EMF measurements to higher temperatures by improving the techniques used for cell preparation. A number of high temperature runs have been partially successful up to about 100°C.

Modifications of our cell preparation techniques have been examined in some detail. We have used ultrasonics to prepare amalgams in situ, to effect more intimate contact between halide and amalgam and to degas the system more effectively when evacuating.

Three half cells were used in the measurements, Zn(Hg)/ZnCl_2 , Cd(Hg)/CdCl_2 , $^{60}\text{NH}_3$ and Tl(Hg)/TlCl . Electrolytic solutions were generally NH_4Cl in NH_3 . A few cells were also tried, with negative results, using sulfates instead of chlorides. It was hoped that the solubility of the ammonium sulfate would become adequate at higher temperatures. No interpretable data were obtainable with the sulfate cells.

Data for chloride cells are tabulated in Tables 9 - 13 and plotted in Figures 7 - 11. The EMFs are of the correct order of magnitude. However, details on the curves have not yet been confirmed by recycling the cell until satisfactory reproducibility is obtained. All cells have failed in the neighborhood of 100°C, that is below the critical point of the solution, for one of two reasons, mechanical failure due to rupture of the cells, or electrochemical failure due to sudden irreversible changes in the EMFs corresponding to open circuits.

One reason for the difficulties at elevated temperatures is that at about 100°C turbulence may occur and become increasingly prominent as the critical point of the solutions is approached.

TABLE 9
POTENTIALS OF CELL NO. 15 (Zn-Cd)

<u>T, °C</u>	<u>EMF, Volts</u>
22	0.4182
24	0.4182
35	0.4144
40	0.4128
50	0.4101
60	0.4107
65	0.4102
70	0.4097
75	0.4081
80	0.4073
85	0.4058
90	0.4053

TABLE 10
POTENTIALS OF CELL NO. 17 (Zn-Cd)

<u>T, °C</u>	<u>EMF, Volts</u>
Room Temperature ($\approx 22^\circ$)	0.3998
61	0.4081
69	0.4047
75	0.4058
80	0.3968
85	0.3952
104	0.3914

TABLE II
POTENTIALS OF CELL NO. 32 (Zn-Cd)

This cell was treated for a few minutes with ultrasonics before being filled with ammonia.

<u>T, °C</u>	<u>EMF, VOLTS</u>
22	0.4305
36	0.4320
45	0.4272
60	0.4244
55	0.4248
50	0.4262
45	0.4281
36	0.4286
45	0.4276
50	0.4223
55	0.4233
60	0.4212
65	0.4216
70	0.4186
75	0.4163

TABLE 12
POTENTIALS OF CELL NO. 22 (Tl-Cd)

<u>T, °C</u>	<u>EMF (BC)</u>	<u>EMF (AC)</u>
23	0.5175	--
30	0.5220	--
35	0.5017*	--
40	0.4842*	--
45	0.4500	0.4675*
50	0.4470*	--
55	0.4410	0.4440
60	0.4329	0.4358
65	0.4250	0.4282
70	0.4149	0.4189
75	0.4071	0.4108
80	0.4151	0.4141
85	0.4060	0.4071
90	0.3934	0.3943
95	0.3792*	0.3737*
100	0.2780	0.2934*
105	0.2058	0.2281*
111	0.1907	0.2129*
95	0.2086	0.2371*

*Very unstable.

TABLE 13
POTENTIALS OF CELL NO. 46 (Zn-Cd)

<u>T, °C</u>	<u>EMF</u>	<u>Specific Conductivity, σ (ohm cm)⁻¹</u>
+ 23	0.4167	0.0438
23	0.4166	
26	0.4155	0.0429
48	0.4093	
51	0.4090	
52	0.4087	0.0372
53	0.4086	
51	0.4084	0.0369
73	0.4037	0.0292
73	0.4032	
80	0.4018	
97	0.3966	
103	0.3941	

5. FUTURE PLANS

In the next quarter, we plan to resolve the turbulence problem at elevated temperatures. Three approaches will be tried. The first is more extensive use of ultrasonics to reduce the tendency for bubble formation. A second approach is to set up the cell so that it can be baked under vacuum prior to filling, the filling also being carried out under vacuum. Again, it is anticipated that this would be very effective in reducing the tendency for bubble formation. The third approach is to prepare concentration cells without liquid junctions and with soluble salts. The advantage here is that the turbulence would not destroy the interfaces between insoluble salts and electrode surfaces.

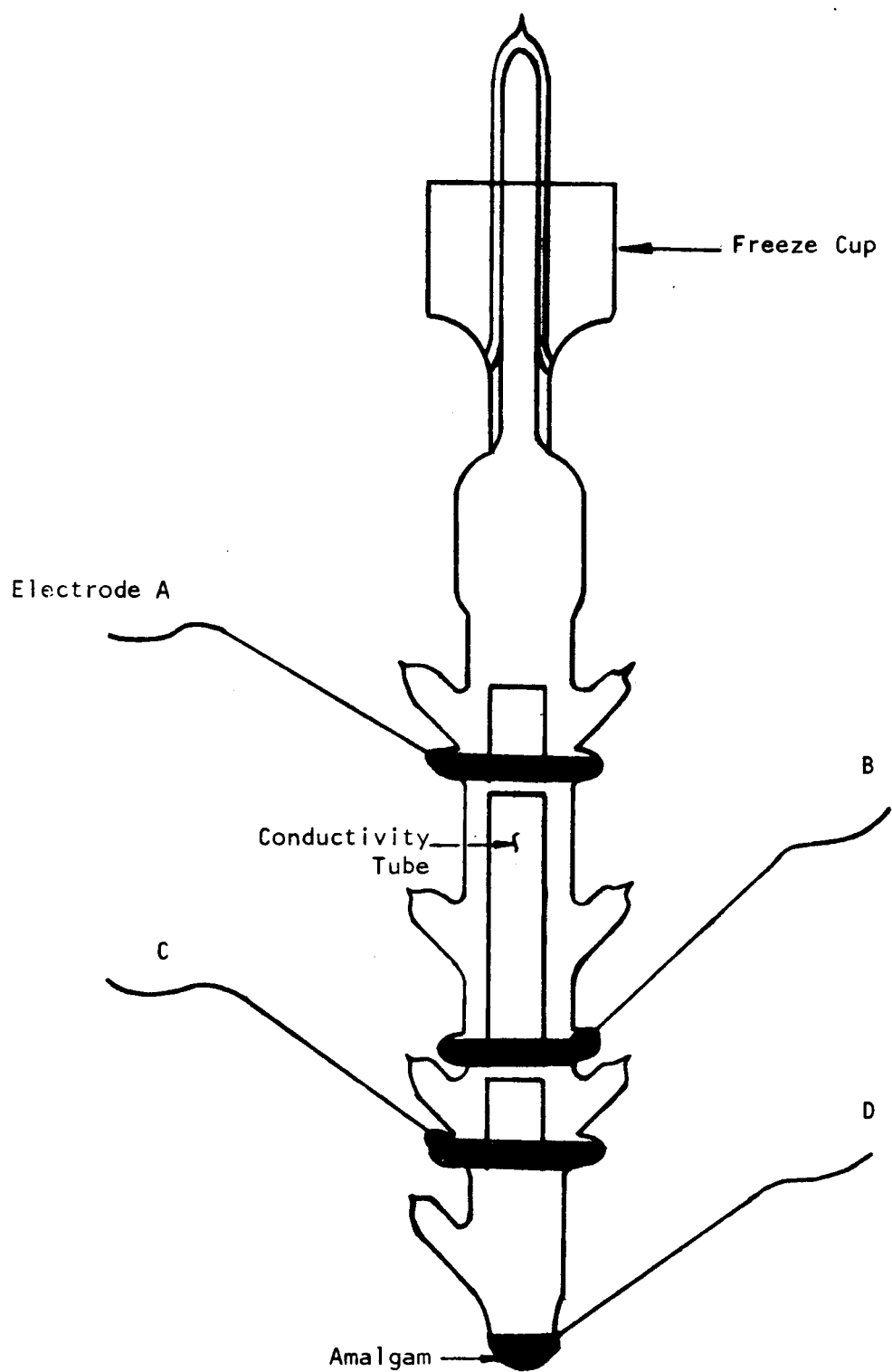


Figure 1. Conductivity Cell

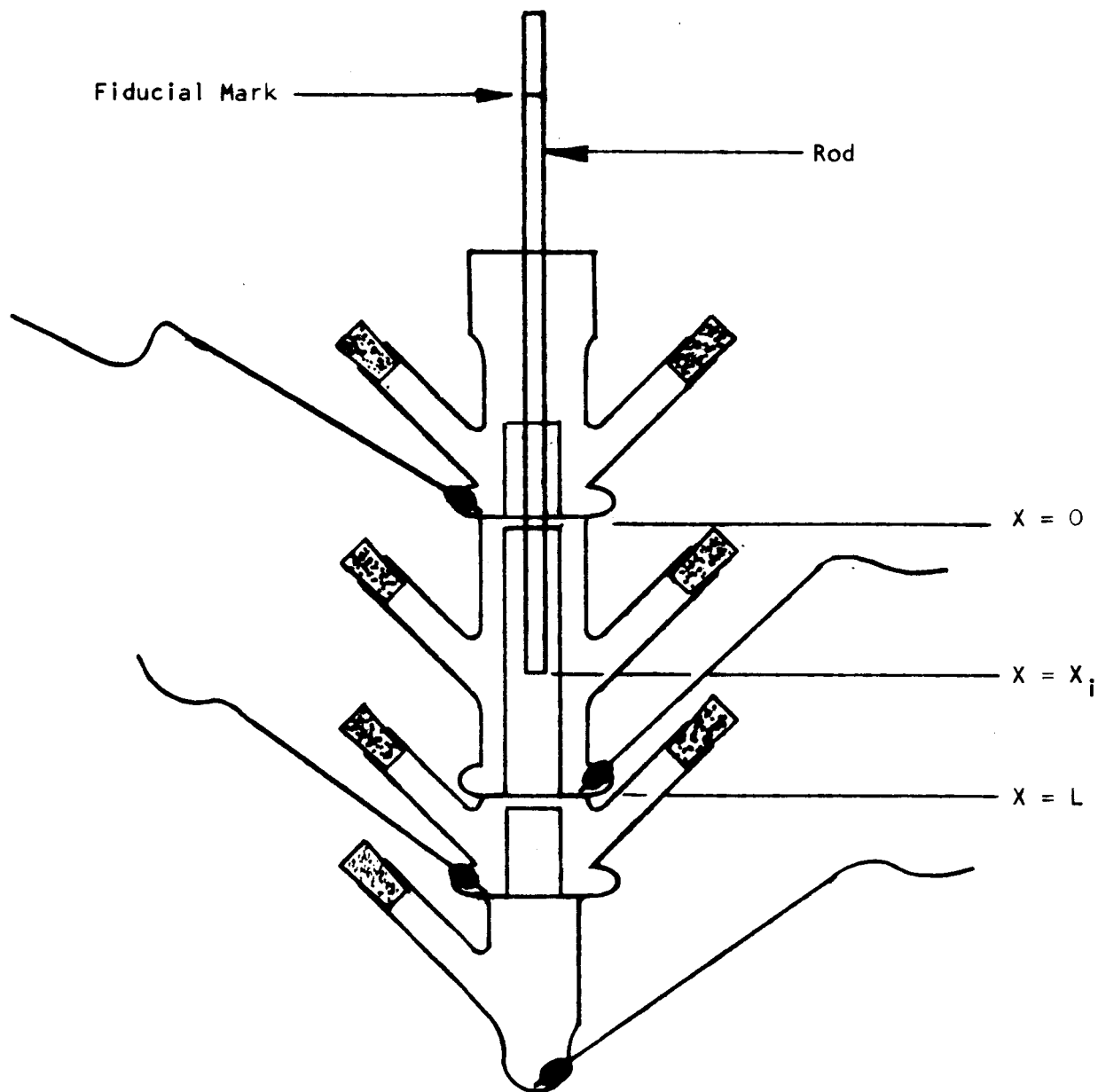


Figure 2. Conductivity Cell with Rod
Inserted in Conductivity Tube

Figure 3. Voltage-Current Plot For Cell No. 3
Containing Mercury

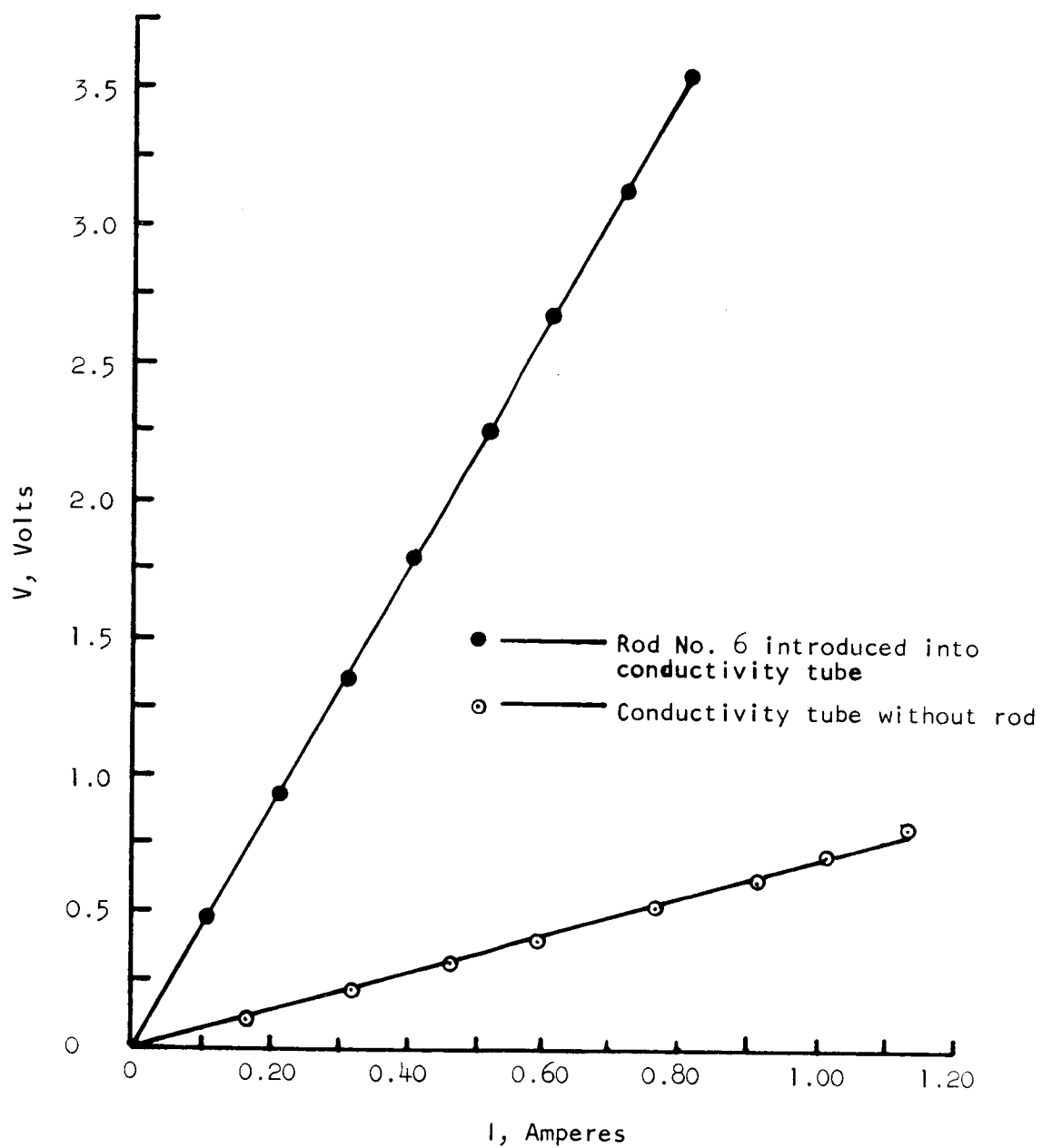
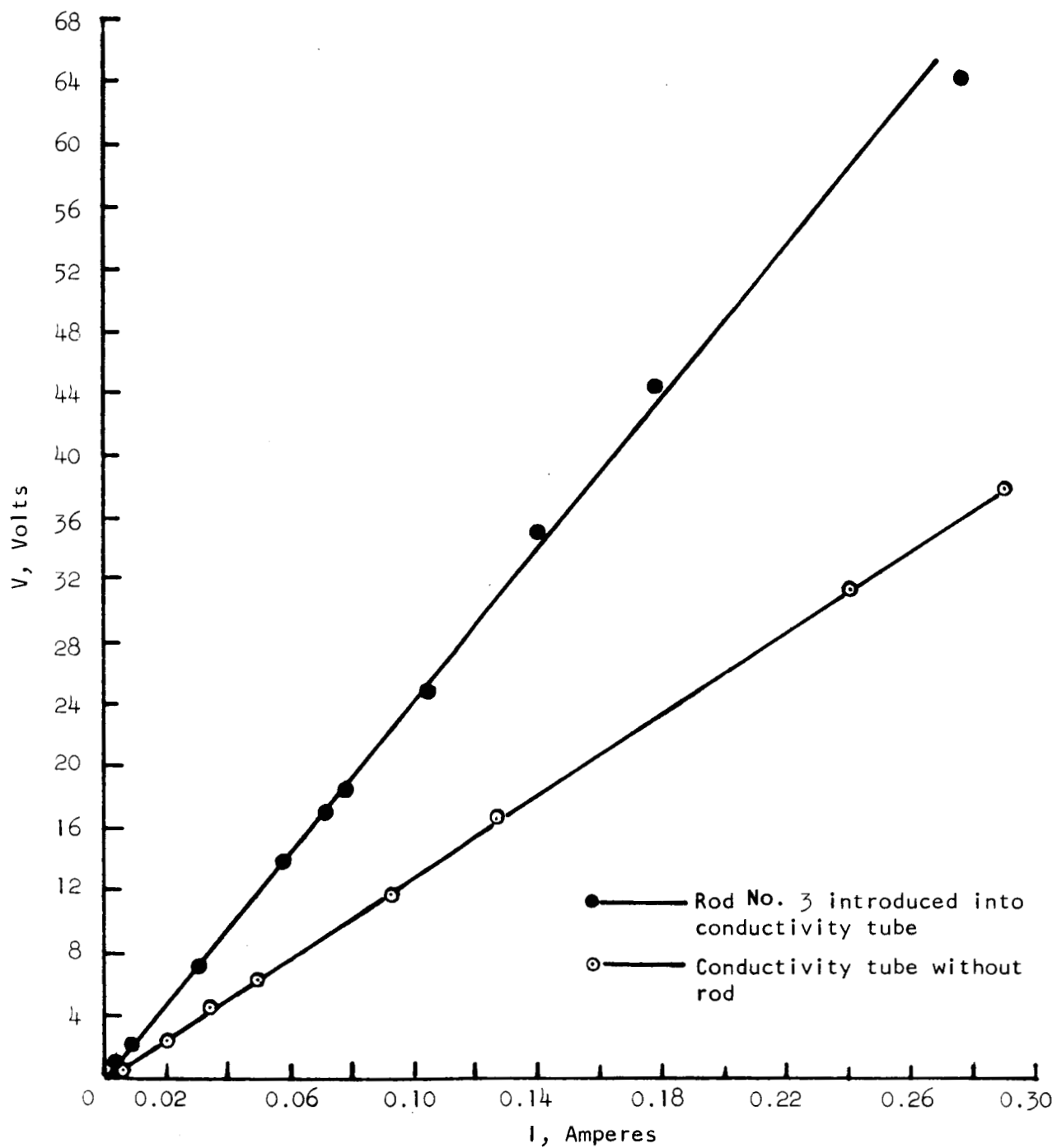


Figure 4. Voltage-Current Plot for
Cell No. 3 Containing 1M KCl



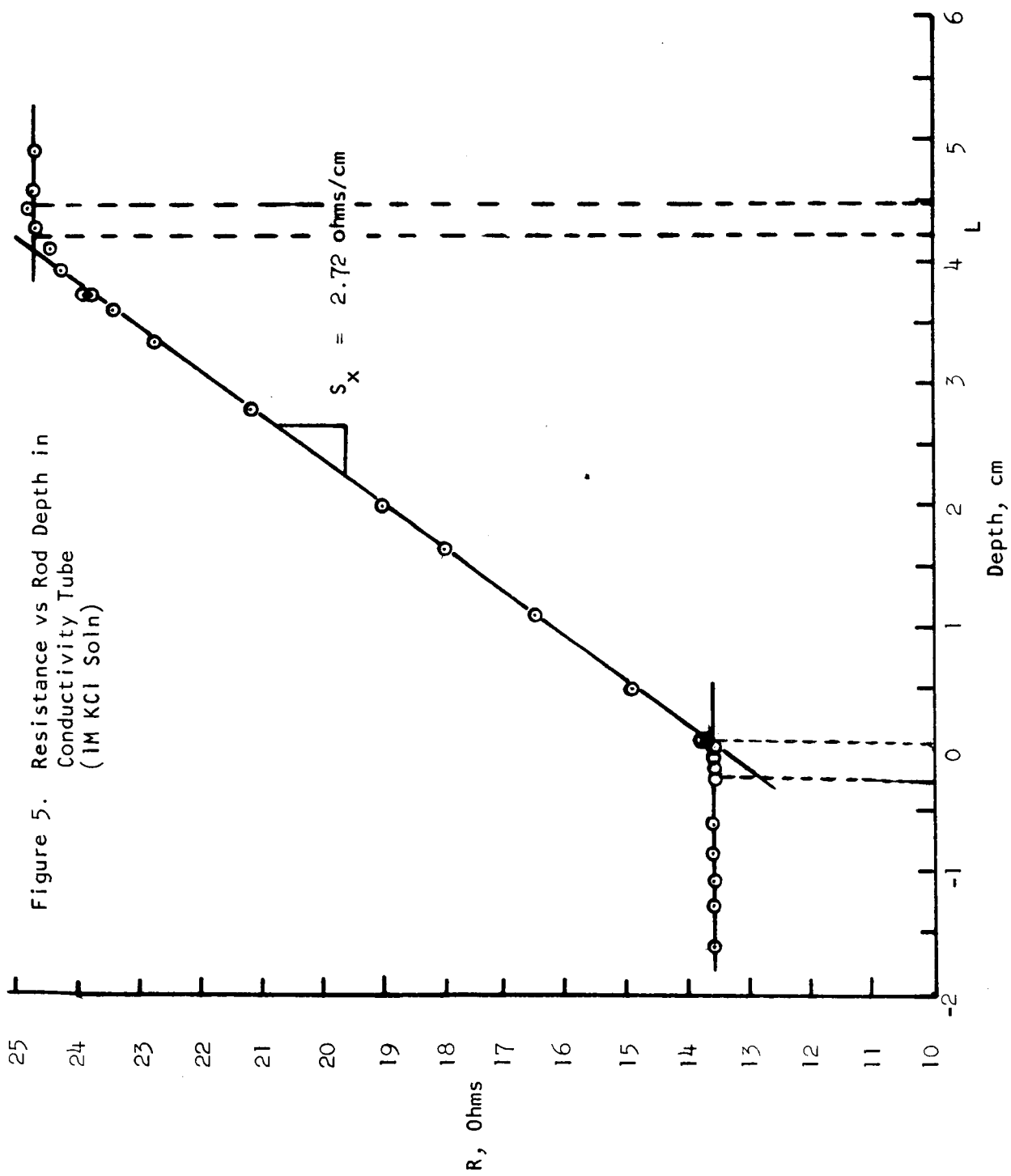


Figure 6. Potential of Cell No. 12
(Zn-Cd)

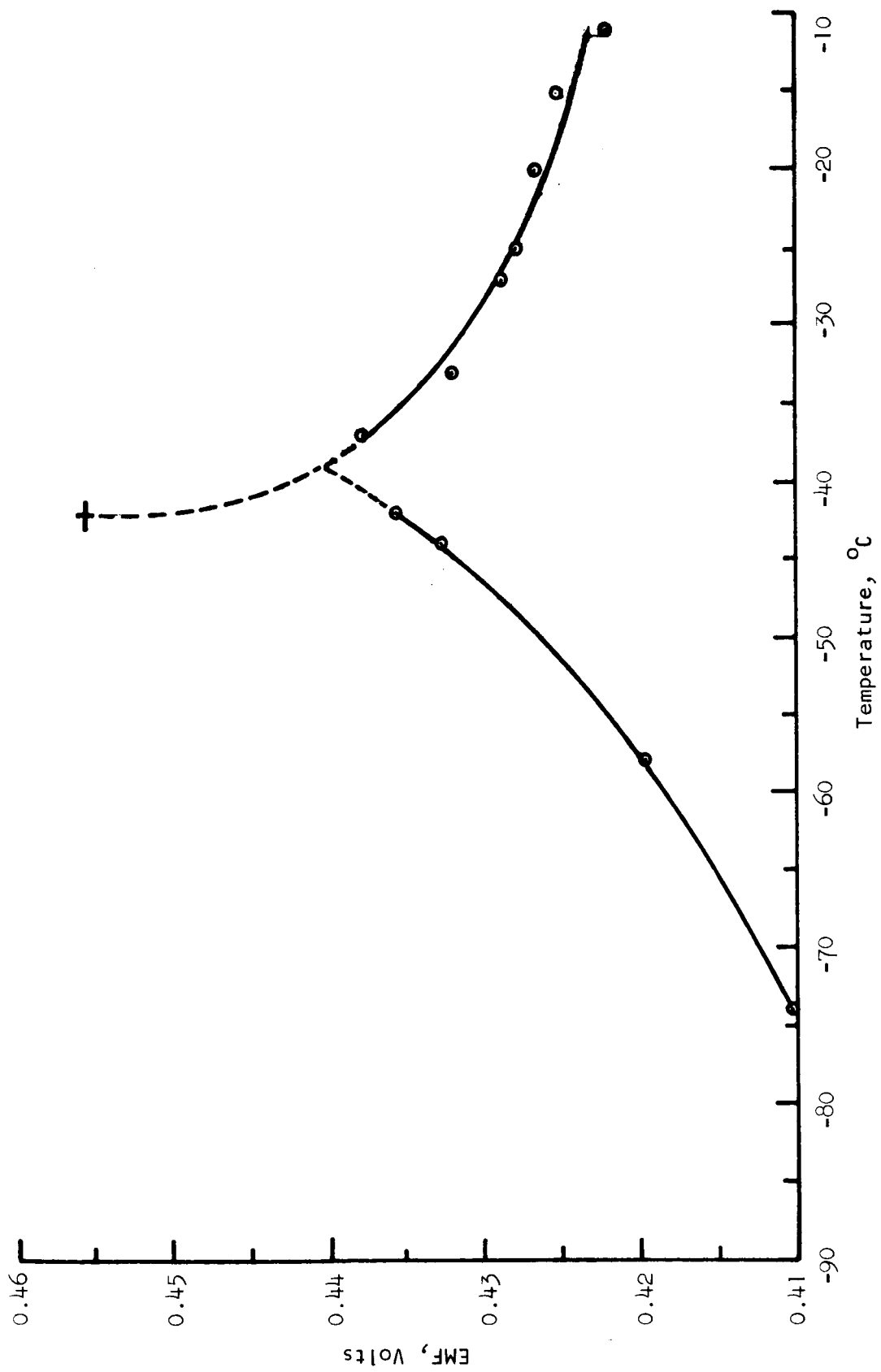


Figure 7. Potentials of Cell No. 15
(Zn-Cd)

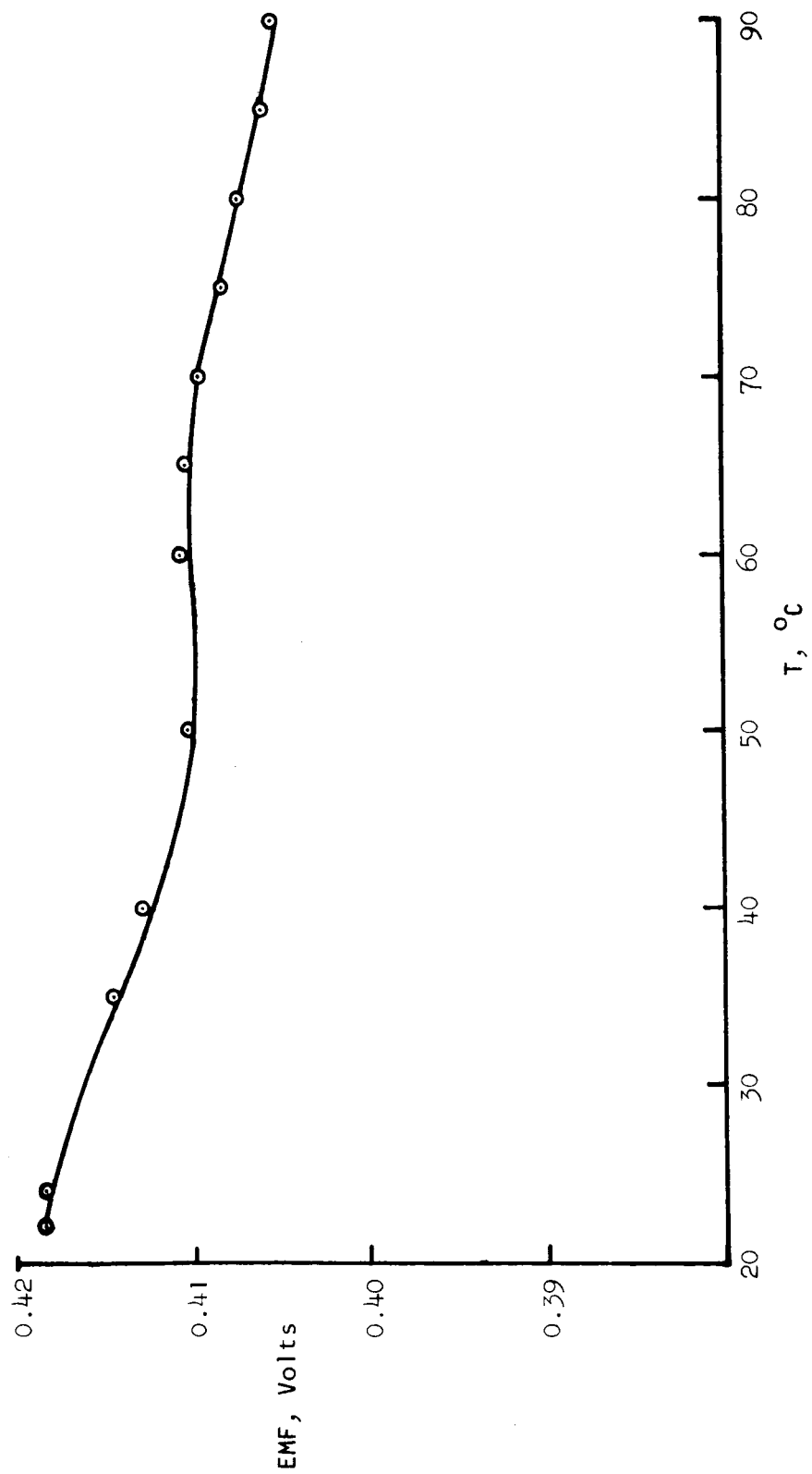


Figure 8. Potentials of Cell No. 17
(Zn-Cd)

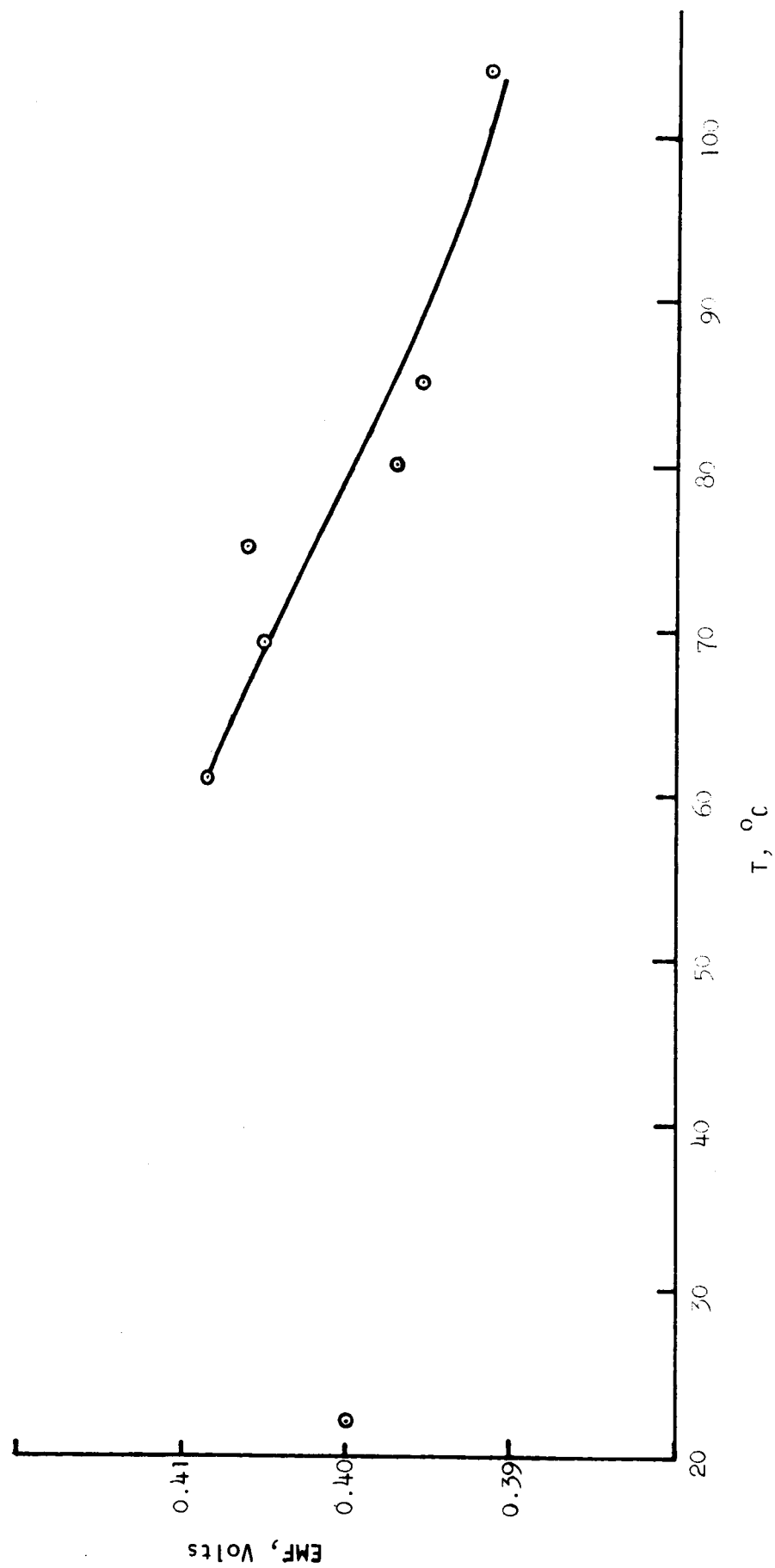


Figure 9. Potential of Cell No. 32
(Zn-Cd)

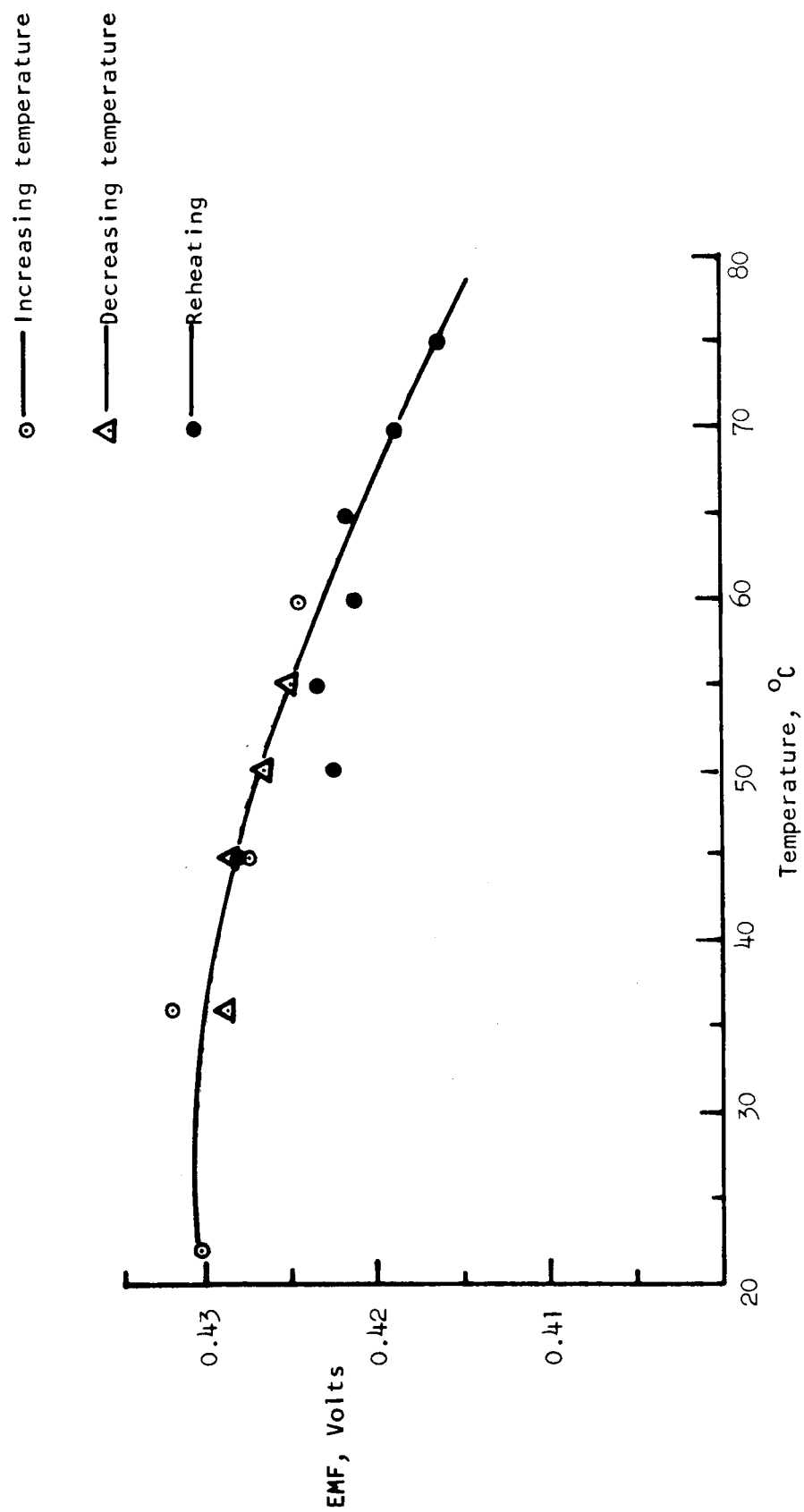


Figure 10. Potential of Cell No. 22
(Tl-Cd)

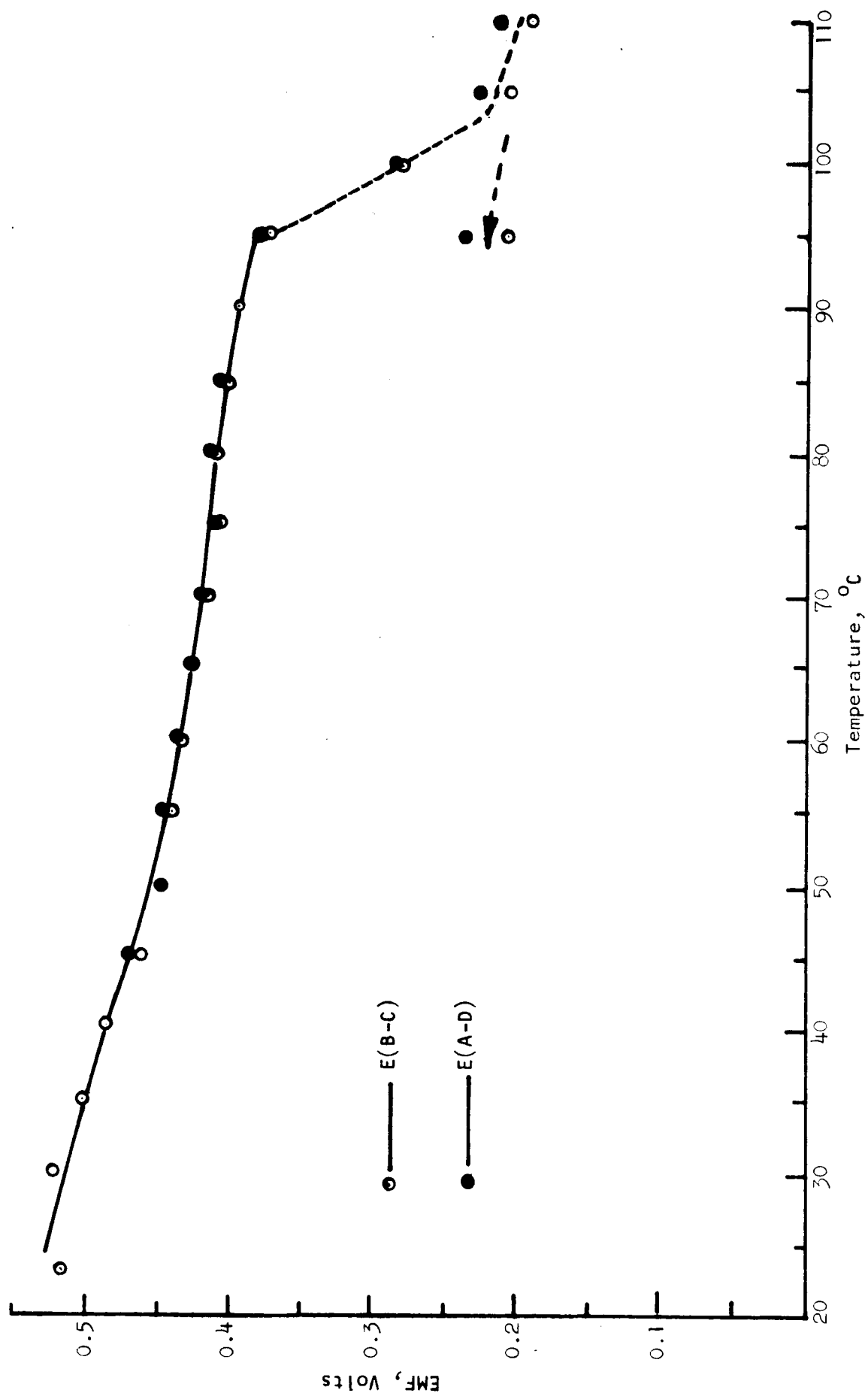
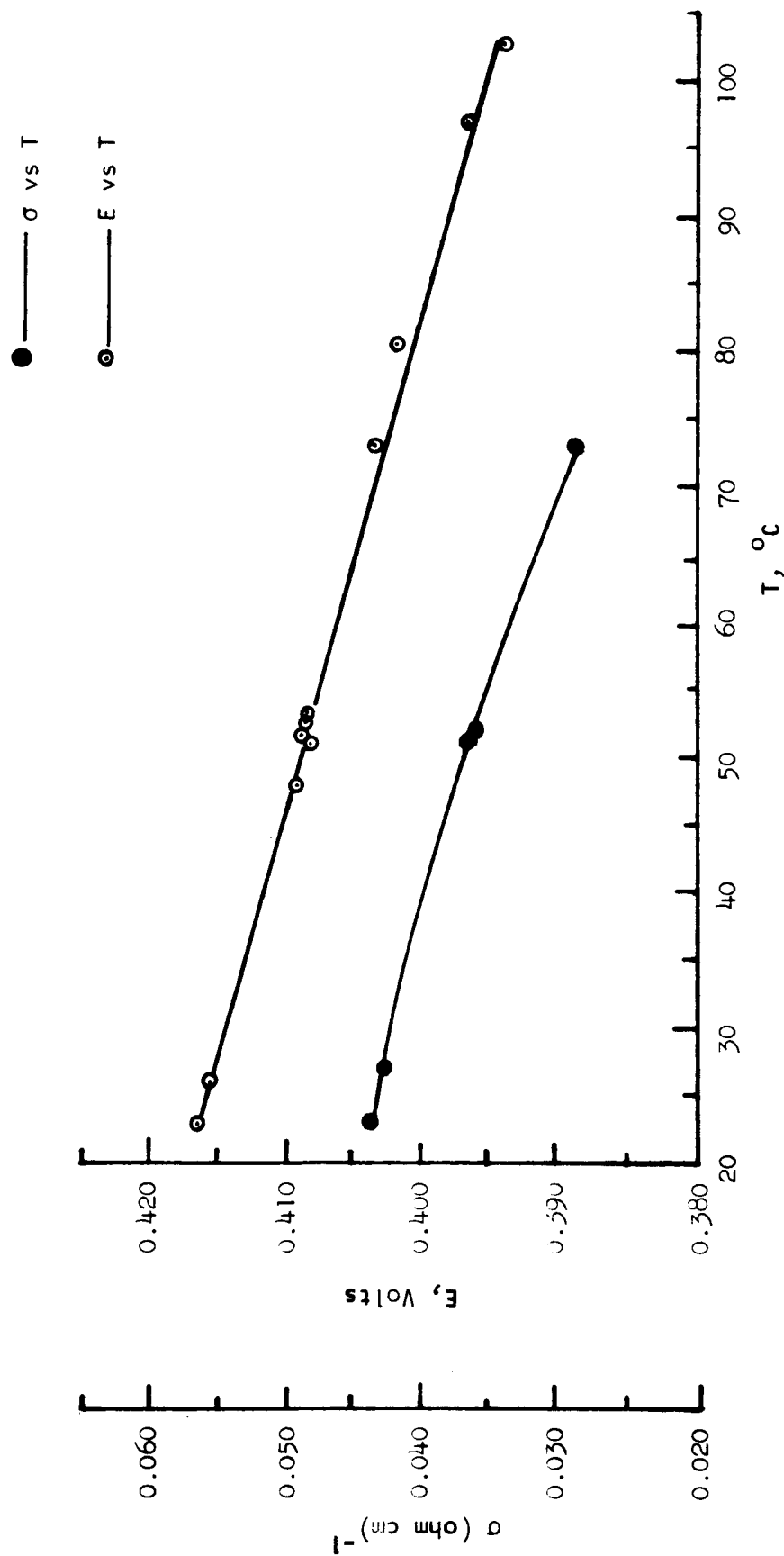


Figure 11. Electrode Potentials and Conductivity
Data for Cell No. 46
(Zn-Cd)



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Attn: Richard Ferguson (for EP-5)
Robert Cohen
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Jet Propulsion Laboratory
4800 Oak Grove Drive
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DEPARTMENT OF THE ARMY

U. S. Army Engineer R and D Labs
Fort Belvoir, Virginia
Attn: Electrical Power Branch

U. S. Army Engineer R and D Labs.
Fort Monmouth, New Jersey
Attn: Arthur F. Daniel (Code SELRA/SL-PS)

Harry Diamond Labs.
Room 300, Building 92
Connecticut Avenue and Van Ness Street, N.W.
Washington, D. C.
Attn: Nathan Kaplan

Army Materiel Command
Research Division
AMCRD-RSCM T-7
Washington 25, D. C.
Attn: John W. Crellin

Natick Labs.
Clothing and Organic Materials Division
Natick, Massachusetts
Attn: Leo A. Spano/Robert N. Walsh

U. S. Army TRECOM
Physical Sciences Group
Fort Eustis, Virginia
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U. S. Army Research Office
Box CM, Duke Station
Durham, North Carolina
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U. S. Army Mobility Command
Research Division
Center Line, Michigan
Attn: O. Renius (AMSMO-RR)

Hq., U. S. Army Materiel Command
Development Division
Washington 25, D. C.
Attn: Marshall D. Aiken (AMCRD-DE-MO-P)

DEPARTMENT OF THE NAVY

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Department of the Navy
Washington 25, D. C.
Attn: Dr. Ralph Roberts/H. W. Fox

Bureau of Naval Weapons
Department of the Navy
Washington 25, D. C.
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Naval Ammunition Depot
Crane, Indiana
Attn: E. Bruess/H. Shultz

Bureau of Ships
Department of the Navy
Washington 25, D. C.
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Naval Ordnance Laboratory
Department of the Navy
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DEPARTMENT OF THE AIR FORCE

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Dayton, Ohio
Attn: James Elsworth Cooper

AF Cambridge Lab.
L. G. Hanscom Field
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OTHER GOVERNMENT AGENCIES

Defense Documentation Center Headquarters
Cameron Station, Bldg. 5
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PRIVATE INDUSTRY

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Bell Laboratories
Murray Hill, New Jersey
Attn: U. B. Thomas/David A. Feder

Boeing Airplane Company
Seattle, Washington
Attn: Henry Oman

Borden Chemical Co.
Central Research Laboratory
P. O. Box 9524
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Burgess Battery Company
Freeport, Illinois
Attn: Dr. Howard J. Strauss

C and D Batteries
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Conshohocken, Pennsylvania
Attn: Dr. Eugene Willihnganz

Calvin College
Grand Rapids, Michigan
Attn: Prof. T. P. Dirkse

Delco Remy Division
General Motors Corporation
Anderson, Indiana
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Post Office Box 290
Joplin, Missouri
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Electric Storage Battery Company
Missile Battery Division
Raleigh, North Carolina
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Electric Storage Battery Company
Carl F. Norberg Research Center
Yardley, Pennsylvania
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